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NONMETALS TEST AND EVALUATION

Delivery Order 0007: The Development of On-Aircraft Surface Preparations Utilizing Sol-Gel Coatings for Adhesive Bonding Aluminum Alloys

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14. ABSTRACT

Current high-performance surface preparations for adhesive bonding of metal alloys typically require the use of acids, volatile organic compounds, and chromated bond primers. These processes are becoming difficult to use due to existing and proposed environmental and health regulations. The Strategic Environmental Research and Development Program funded a Tri-Service team to develop prebond surface preparations for aluminum, titanium, and steel using sol-gel technology. As part of that effort, UDRI assisted in the development and optimization of on-aircraft aluminum surface preparations based on two sol-gel chemistries, the Boeing Company's Boegel-EPII and Chemat Technologies, Inc.'s AL 9201. Several experiments were conducted to evaluate surface activation steps prior to sol-gel application, process factors, and interactions that were required to define process-operating windows for surface preparation steps. Key steps investigated included surface activation and sol-gel application as well as postapplication drying and priming. Surface activation steps using Boegel-EPII included grit-blast, nylon pad abrasion, sandpaper abrasion, and laser deoxidation. Chemat AL 9201 was evaluated using grit-blasting. Resulting processes were environmentally friendly, robust, and quick. In addition, processes yielded initial bond strength and long-term environmental durability performance that met or exceeded performance of existing processes such as grit-blast/silane phosphoric acid containment system, and sulfuric acid-sodium dichromate paste etches.

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PREFACE

This report covers work performed by the University of Dayton Research Institute (UDRI), Dayton, Ohio 45469-0138, during the period from January 1998 to May 2001. All work was performed under Air Force Contract Number F33615-95-D-5616, Delivery Order 0007. The work was administered under the direction of the Systems Support Division of the Air Force Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. Gregory Elam and Lt. Heather Crooks were the Contract Monitors. Mr. James J. Mazza was the government engineer and technical leader. The UDRI Program Manager was Robert Askins. The Principal Investigator was Mr. Daniel McCray. Technical support was provided by Mr. Jeffrey Smith of UDRI as well as Ms. Kylie Huber and Ms. Carly Wreesman from the Southwestern Ohio Council for Higher Education (SOCHE).

This research was partially funded by the Strategic Environmental Research and Development Program (SERDP) under Project PP-1113 and is a part of the overall PP-1113 effort. The Boeing Company led the sol-gel chemistry development and technical integration associated with this effort. The United States Air Force, Navy, and Army led the process development efforts for aluminum, titanium, and steel alloys, respectively.

Thanks are given to Dr. Kay Blohowiak for sol-gel chemistry development and advice throughout this program. All PP-1113 team members are thanked for their contributions, particularly the Navy and Army leads, Georgette Gaskin of NAVAIR-Patuxent River, MD, and William DePiero of the TACOM-ARDEC Armament Materials Team at Picatiny Arsenal, NJ. Appreciation is also extended to Mr. Jay Fiebig and Mr. Bill Schweinberg of WR-ALC/TIEDD at Robins AFB, GA, for providing valuable on-aircraft bonded repair expertise throughout this program.

1 BACKGROUND

High-performance surface preparations for adhesive bonding of metals typically require the use of strong acids or bases, volatile organic compounds (VOCs), and hexavalent chromium. Surface preparations used for on-aircraft repair of aluminum rely on hazardous materials or inconvenient processing steps, or they do not yield adequate bond performance. Grit-blast/silane (GBS)[1], phosphoric acid containment system (PACS)[2], which is a version of phosphoric acid anodize (PAA)[3], and certain acid paste etches are the high-performance surface treatments currently available for on-aircraft application. All are used in conjunction with chromated, high-VOC primers. All are time consuming for on-aircraft repair. Furthermore, their use is becoming more difficult due to existing and proposed environmental, safety, and health regulations.

The Strategic Environmental Research and Development Program (SERDP) funded a Tri-Service team to optimize sol-gel coatings for aluminum, titanium, and steel prebond preparation in order to reduce the environmental impact of the above-mentioned hazardous materials. The effort involved development of processes based on two sol-gel chemistries: (1) the Boeing Company's Boegel-EPII[4] and (2) Chemat Technologies, Inc. AL 9201[5]. UDRI, under AFRL contract F33615-95-D-5616, Delivery Order 0007, evaluated, developed, and optimized on-aircraft repair bonding processes for aluminum utilizing both sol-gel chemistries.

Goals of the AFRL/MLSA and UDRI effort were to establish prebond surface preparation processes that provided similar bond performance to PAA that could be successfully performed on aircraft at the field and depot level. Emphasis was placed on reducing hazardous materials associated with adhesive bonding, such as VOCs and hexavalent chromium, while reducing repair time by eliminating or reducing the number of required elevated temperature thermal cycles. Development of repair processes using the Boegel-EPII was similar to the approach used by AFRL/MLSA when optimizing GBS. The more reactive sol-gel chemistry allowed for the possibility of using several simple, inexpensive deoxidation/surface activation processes in addition to grit-blasting. These alternatives were not viable for GBS.

2 TEST PLAN

2.1 Test Materials

All tests performed in this program used bare Al 2024-T3 or Al 7075-T6 adherends. Cytec BR 6747-1 waterborne, chromated bond primer was applied to adherends, unless otherwise noted. The primer was applied with a Binks 105 touch-up spray gun to a cured thickness of 0.1-0.3 mil (0.0001-0.0003 inch). Primer was dried at ambient temperature (70°F) for 30 minutes and cured according to the manufacturer's recommendations unless otherwise noted.

3M Company AF 163-2M (0.06 psf) epoxy film adhesive was used for all adhesive bonding, unless otherwise noted. The adhesive was applied to the adherends and cured according to the manufacturer's recommendations of 60 minutes at 250°F under 35-40 psi unless otherwise noted.

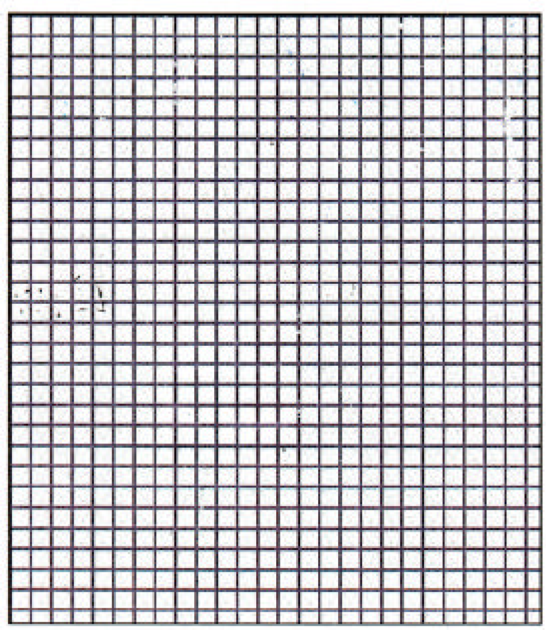
2.2 Wedge (Crack Extension) Test

Adhesive bonds to metallic structure are susceptible to degradation due to moisture attack at the metal interface. Therefore, the resistance to moisture degradation is the key factor in determining the quality and longevity of a bonded joint. The prebond surface preparation is the determining factor for bond moisture durability performance. Properly treated surfaces resist moisture degradation while improper surface treatments more readily lead to degradation and interfacial failures. The wedge test (ASTM D 3762)[6] was used to evaluate the durability of bonded joints during optimization of surface preparation processing steps during this program.

For the wedge test, 0.125-inch aluminum sheet stock was sheared into 6.5-inch square adherends (Section 2.3). Upon completion of the surface preparation and bonding cycle, wedge test panels were machined into five 1-inch wide specimens using a gang-cutting mill. The bondline thicknesses (BLT) of all wedge test specimens were measured with the use of an optical microscope. Stainless steel wedges were driven into the end of the wedge test specimens using a hammer, and the initial crack lengths were measured. Wedge test specimens were aged at 120°F and 95-100% relative humidity (RH) or 140°F and 95-100% RH. Crack growth measurements were taken after 1 hour, 8 hours, 24 hours, 7 days, 14 days, 21 days, and 28 days.

After 28 days in humidity, specimens were split apart to determine the failure modes. Failure modes were considered to be either cohesive or adhesive. Cohesive failures were those that failed within the adhesive. All remaining failure modes were designated as adhesive, including those within the adhesive primer and at the various interfaces between bond constituents. Often, both types of failure occur within the same specimen. Therefore, failure modes were recorded as percentages of cohesive failure, with 0% cohesive failure representing complete adhesive failure. Failure mode percentages were determined using the grid shown in Figure 1. The grid was produced by Boeing (as part of the SERDP PP-1113 effort) and printed on a standard overhead transparency sheet. It was placed over the specimen and the total number of squares in the test area was counted. Then, the amount of squares with cohesive failure was counted. The percentage of cohesive failure was calculated by dividing the number of cohesive squares by the total number of squares in the test area. The test area was defined as the area between the initial crack and final crack. This area was typically a lighter shade of the adhesive color than the rest of the test area due to high stresses. All wedge data presented in this report are the average of five specimens from the same wedge panel unless otherwise noted.

Figure 1: Grid Used to Determine Failure Modes for Wedge Test Specimens

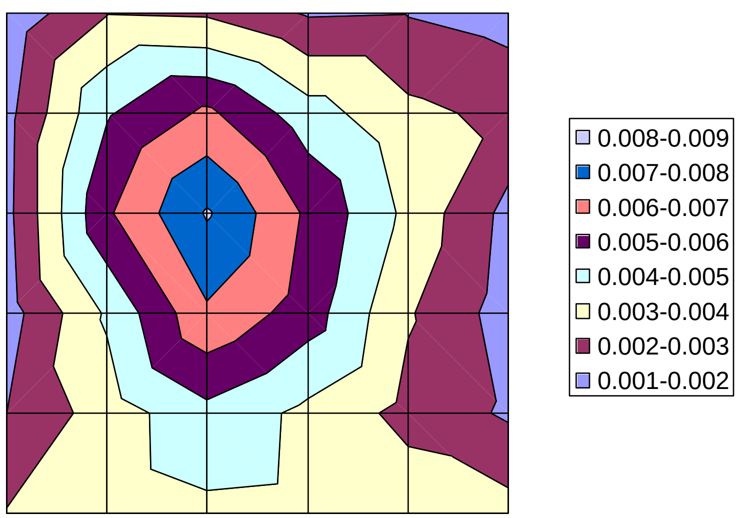


Acceptable or passing results in the wedge test were defined by AFRL/MLSA as cohesive failure modes after 28 days at environment. Although this typically requires 100% cohesive failure, an exception was made since the sol-gel surface preparations exhibited small “nicks” of interfacial failure at the edges of the specimens. Failure modes that exhibited small “nicks” of interfacial failure occurring only at the edges of the specimens were considered acceptable as long as the total area of the interfacial failure was 5% or less.

2.3 Bondline Thickness Control

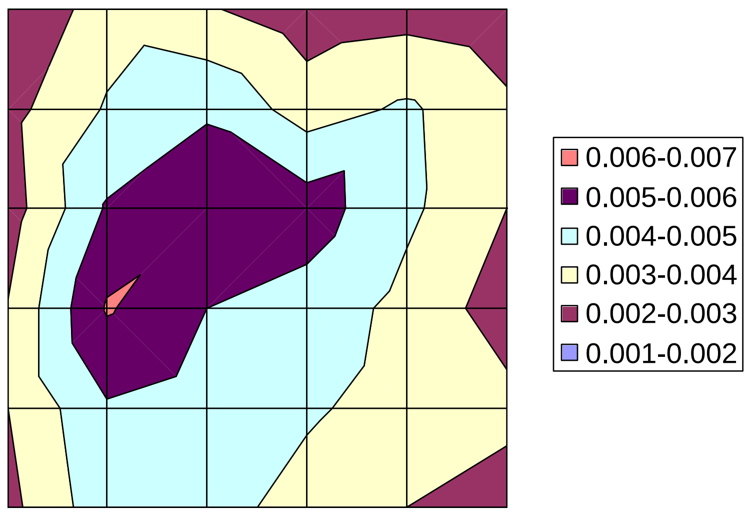
In order to obtain consistent results throughout this program, a uniform bondline thickness of 5.0 mils (0.0050 inch) $\pm$1 mil was desired for each specimen. 3M Company AF 163-2M is manufactured with a mat carrier cloth embedded in the film to enable bondline thickness control. In order to verify the bondline thickness across an entire wedge test panel, a standard wedge test panel (6-inch square) was bonded with 0.06 psf AF 163-2M and sectioned into 1-inch x 1-inch samples. Sample edges were polished, and bondline thicknesses were measured with an optical microscope and plotted to determine the bondline variation across the entire panel. Results of the baseline bondline thickness panel are shown in Figure 2. The edges of the wedge test panel had much thinner bondlines (0.001-0.002 inch) than the center (0.007 inch). Not only did all specimens fail to meet the desired bondline thickness of 0.005 inch, but the bondlines were very uneven across the panel.

Figure 2: Bondline Thickness Variation in Standard Wedge Test Panel Bonded with 0.06 psf AF 163-2M



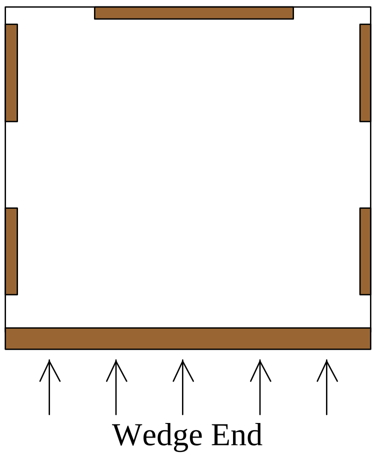
A second wedge test panel was bonded with 3M Company AF 163-2K (knit carrier) with a thicker adhesive weight (0.085 psf) to determine if the thicker film of adhesive would exhibit similar results. The results (Figure 3) show the 0.085 psf adhesive provided more uniform bondlines than the 0.06 psf adhesive under identical processing conditions, but the bondlines were still uneven across the panel and too thin at the edges (0.002-0.003 inch).

Figure 3: Bondline Thickness Variation in Standard Wedge Test Panel Bonded with 0.085 psf AF 163-2K



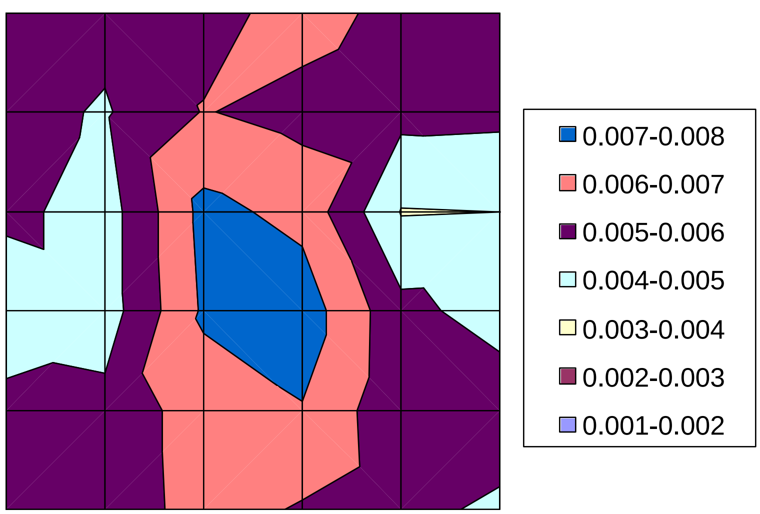
Several steps were taken to obtain more even bondlines within the desired bondline thickness range of 0.005-inch $\pm$1 mil. It was felt that the flexible bladder in the portable autoclave used to apply heat and pressure to cure the wedge test panels in this program, was pinching the edges of the wedge test panels causing the adhesive to flow from the edges to the center of the panel. Therefore, the size of the wedge test panel was increased from the ASTM standard 6-inch square, to 6.5-inch square. During the application of adhesive to the larger panel, thin strips of 0.003-inch thick Teflon tape were placed in the bondline around the panel perimeter (Figure 4) to prevent the flexible bladder from squeezing the adhesive to the center of the panel. This tape was only applied to the edges of the panel and was removed from the panel during machining of specimens. Results of the bondline thickness measurements from a wedge test panel bonded using the tape shims and 0.06 psf AF 163-2M are shown in Figure 5. The bondline thickness was more uniform and closer to 0.005 inch. Therefore, all wedge test panels in this program were fabricated with 6.5-inch square adherends and shimmed around the edges with 3-mil tape as shown in Figure 4.

Figure 4: Alternate 6.5-Inch Square Wedge Test Panel Shimmed with 3-Mil Tape at the



Edges

Figure 5: Bondline Thickness Variation in Alternate 6.5-Inch Wedge Test Panel Shimmed with 3-Mil Tape at the Edges and Bonded with 0.06 psf AF 163-2M



3 EFFECT OF PROCESS VARIABLES USING BOEGEL-EPII SOL-GEL SOLUTION

For metals, prebond surface preparation is the key factor determining the durability of an adhesive bond. Proper surface preparation provides a suitable interface between the metal substrate and adhesive that will withstand environmental factors such as heat and moisture. Surface preparations typically begin by removing contaminants and weak boundary layers or chemically incompatible layers through degreasing and mechanical abrasion steps. The surface preparation should then create a stable and chemically compatible interface for the adhesive to bond to the metal.[7] To provide this interface, Boeing Phantom Works developed and optimized Boegel-EPII sol-gel solution in prior work with the US Air Force[8]. Boegel-EPII is a waterborne solution composed of four separate components that must be mixed prior to use. The four components are deionized water, silane coupling agent, a zirconium compound, and acetic acid[9]. The standard Boegel-EPII mixing instructions are shown in Table 1. Adhesive bonding for this project using Boegel-EPII chemistry followed a few general steps, as shown in Figure 6 Figure 6. Process variables associated with the processing steps were evaluated and determined to be significant or insignificant to the bond performance. Significant processing steps were defined with windows of performance. Overall, the processing steps began with a degreasing step designed to remove organic contamination from the bond surface. This was followed by deoxidation and surface activation by one of several methods:

- grit-blast with 50 micron aluminum-oxide grit,
- abrasion with nylon abrasive pads,
- abrasion with sandpaper, and
- laser treatment.

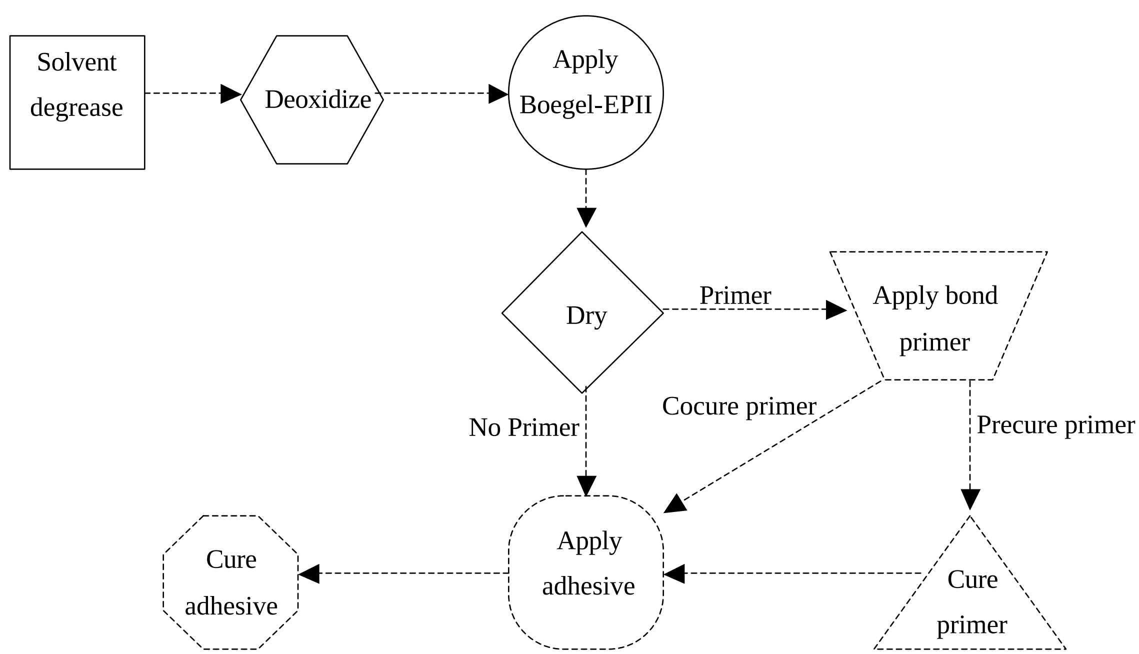
After deoxidation, Boegel-EPII was applied and dried at ambient temperature (typically 70°F ±5°F). Depending on the process, sol-gel treatment was followed by application of bond primer prior to bonding, or bonding proceeded immediately without primer. When used, primer was precured according to the manufacturer's recommended instructions or cocured with the AF 163-2M adhesive after ambient temperature drying, unless otherwise noted. Once adhesive was applied, the panels were cured in a portable autoclave.

A large amount of data were generated during this project by varying deoxidation/surface activation steps during the surface preparation including those associated with precleaning, surface activation, application of Boegel-EPII solution, drying, and priming (if necessary). Additionally, variables included choice of materials, processing windows, and time between steps. Data will be presented in sections according to the surface activation method.

Table 1: Boegel-EPII Standard Mixing Instructions

Step	Chemicals	Procedure
1	5 mL n-Zirconium Propanol (TPOZ) 2.25 mL Glacial Acetic Acid (GAA)	Mix TPOZ & GAA in small vile. Agitate until fully mixed. Mixture should warm since reaction is exothermic. Let sit for 10-15 minutes.
2	10 mL γ-glycidoxypropyltrimethoxysilane (GTMS) 500 mL deionized water	Mix GTMS and water in a flask. Mix with magnetic mixer.
3	GAA & TPOZ mixture GTMS & water mixture	After 10-15 minute time has elapsed, pour GAA / TPOZ mixture into GTMS / water mixture.
4	Boegel EPII sol-gel solution	Mix with magnetic mixer for a minimum of 30 minutes. Boegel EPII solution must be used within 10 hours of initial mixing.

Figure 6: Sol-Gel Surface Preparation General Process Diagram



3.1 Optimization of Processing Parameters Using Boegel-EPII with Grit-Blast Surface Activation

Grit-blasting is a key component of many metal surface preparation processes. Grit-blasting removes existing oxide layers while creating a rough surface morphology that is conducive to bonding. Grit-blasting provides excellent durability and initial strength results when used with the silane coupling agent. In addition, organic contaminates are easier to detect optically on grit-blasted surfaces, thus providing a good quality control measure during the surface preparation. Experience obtained during GBS development led to the choice of grit-blasting as a logical surface activation step for use with Boegel-EPII to obtain desired bond performance.

In order to optimize the process parameters involved with activating the bonding surface and subsequent application of Boegel-EPII and bond primers, a designed experiment was conducted to determine significant processing factors. Once identified, several smaller experiments were conducted to determine optimum operating windows for individual steps in the surface preparation.

3.1.1 Grit-Blast Designed Experiment

Several key processing factors for a grit-blast/sol-gel process using Boegel-EPII were evaluated via a designed experiment was conducted using an L16 array. The evaluated processing factors are listed in Table 2. Aluminum alloy type, Boegel-EPII application method, Boegel-EPII wet time, Boegel-EPII dry method, Boegel-EPII dry time, and bond primer factors were all assessed using a matrix consisting of 16 wedge test panels.

Table 2: Grit-Blast Sol-Gel Designed Experiment Processing Parameters

Factor	Parameter #1	Parameter #2
Alloy	Al 2024-T3	Al 7075-T6
Boegel-EPII application method	Spray	Brush
Boegel-EPII wet time	10 minutes	20 minutes
Dry method	nitrogen force	ambient dry
Dry time	1 hour	4 hours
Primer	BR 6747-1	none

Wedge test adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny surface was obtained. The nylon pad abrasion step was used to generate a baseline surface from which to start the process each time. Adherends were then blasted with alumina grit having a nominal size of 50 μm. Clean, dry, compressed nitrogen was used to grit-blast the panels in order to prevent contamination from oil, condensed moisture, and other possible contaminants. After the panels were grit-blasted, compressed nitrogen (35 psi) was used to remove as much residual grit from the surface as possible. The sol-gel processing factors were then varied according to Table 2.

Panels were brush coated with the Boegel-EPII solution for the allotted wet time. During the wet time, the panels were visually inspected for water breaks. All panels exhibited a water-break-free surface during sol-gel application. Panels were then force-dried with compressed nitrogen or allowed to ambient dry for a given time. After the dry time had elapsed, panels were primed with Cytec BR 6747-1, when specified, and bonded with AF 163-2M. Primer was applied to a nominal thickness of 0.1-0.3 mil (0.0001-0.0003 inch).

Panels were machined into 1.0 inch-wide wedge test specimens. The bondline thickness of each specimen was measured with an optical microscope. The specimens were then tested at 140°F and 95-100% RH. The failure modes (percentage of cohesive failure) of the specimens were used to calculate the significance of each factor using the design of experiments philosophy[10]. Anova analysis was performed on the results and a chart was plotted in order to distinguish the significance of each factor and interaction. All factors and interactions with a standardized effect greater than the 95% confidence limit were considered significant, as shown in Figure 7. Only the primer and sol-gel dry method factors were considered to be significant. The primer/dry method interaction was also significant. Best durability was detected in panels when they were force-dried with nitrogen and primed. All processing factors other than primer and dry method were considered insignificant, including sol-gel application method, sol-gel wet time, and sol-gel dry time. Most wedge test specimens with optimum processing conditions exhibited crack growth less than 0.25 inches with cohesive failure modes (within the adhesive layer) as shown in

Figure 8. Although the failure modes were primarily cohesive in nature, small "nicks" of adhesive failure (at the metal interface) were detected at edges of many specimens. However, these nicks are difficult to detect visually in Figure 8 due to the small size. Overall, it was estimated that the area of these small nicks was roughly 5% or less of the specimen test area.

Figure 7: Significance of Processing Factors and Interactions for Grit-Blast/Sol-Gel Surface Preparation

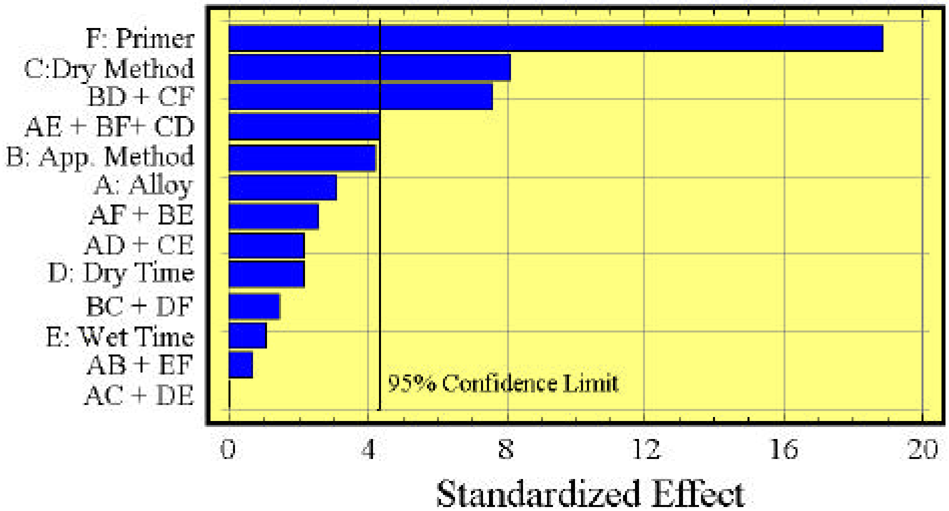
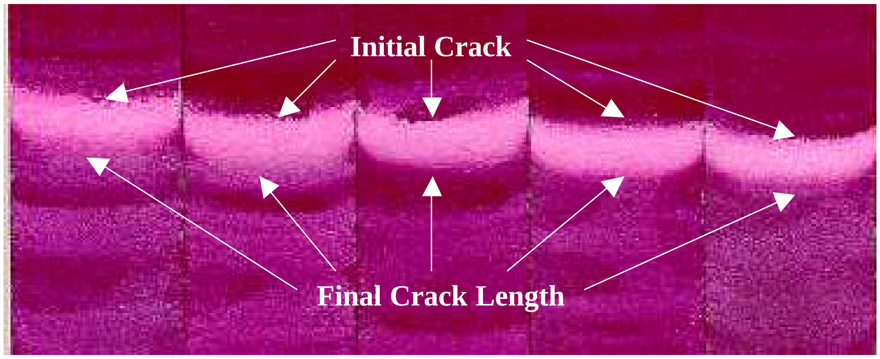


Figure 8: Cohesive Failure Mode Exhibited by Grit-Blasted Boegel-EPII Specimens with Optimum Processing Conditions



3.1.2 Effect of Aluminum Alloy

Although the grit-blast designed experiment (3.1.1) was unable to detect a significant difference between the performance of Boegel-EPII over grit-blasted surfaces on different alloys, a secondary experiment was conducted to verify this result. Grit-blasted wedge test panels were fabricated with adherends composed of bare Al 2024-T3, clad Al 2024-T3, and bare Al 7075-T6 and treated with Boegel-EPII solution to determine if a change in alloy would affect wedge test results.

Wedge test adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50μm aluminum-oxide grit then blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, with the surfaces kept wet for 10 minutes prior to being blown dry with compressed nitrogen (35 psi). Adherends were primed with BR 6747-1 immediately after the panels were blown dry. Primed adherends were dried at ambient temperature (70°F) for 30 minutes and cured for 60 minutes at 250°F. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 140°F and 95-100% RH. Results are show in Table 3. There appears to be no appreciable difference in either crack growth or failure mode due to alloy type.

Table 3: Effect of Alloy Type on Grit-Blast Wedge Test Results

Alloy	**Initial (in)**	**Cummulative Crack Growth (in)**						**Failure Mode***
		1 hr	**8 hr**	**24 hr**	**7 days**	**21 days**	**28 days**	
Al 2024-T3 Bare	1.09	0.04	0.06	0.10	0.16	0.22	0.23	95% co
Al 2024-T3 Clad	1.22	0.01	0.07	0.11	0.15	0.23	0.24	96% co
Al 7075-T6 Bare	1.15	0.07	0.10	0.14	0.20	0.24	0.24	94% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.3 Boegel-EPII Wet Time Evaluation

In order to determine the effect of different Boegel-EPII wetting times, two experiments were conducted varying the Boegel-EPII wet time over grit-blasted surfaces, one with Al 2024-T3 and another with Al 7075-T6. When wet time was evaluated in the grit-blast designed experiment (3.1.1), data showed that the factor was insignificant for wet times of 10 minutes and 20 minutes, so all times between were also considered to be insignificant. The goal of the wet-time experiments was to define the minimum Boegel-EPII wet time required to provide cohesive failures in the wedge test. Wet times between 2 and 12 minutes at 2 minute intervals were evaluated in this experiment.

Wedge test adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50μm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for the specified times. In order to assure that each panel was wetted for only the specified time, the panels were blown dry with compressed nitrogen once the wet time had elapsed. Adherends were primed with BR 6747-1 immediately after the panels were blown dry. Primed adherends were dried at ambient temperature (70°F) for 30 minutes and cured for 60 minutes at 250°F. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 140°F and 95-100% RH.

3.1.3.1 Al 2024-T3 Boegel Wet Time Evaluation

Results of the wet time evaluation on Al 2024-T3 are shown in Table 4. When varying the wet time between 2 minutes and 12 minutes, there appears to be no difference in either the crack growths or failure modes. Since wet times of 20 minutes were evaluated in the grit-blast designed experiment, there is sufficient data to show that wet times from 2-20 minutes provide acceptable wedge test results on Al 2024-T3 grit-blasted surfaces.

Table 4: Effect of Boegel Wet Time on Al 2024-T3 Grit-Blast Wedge Test Results

Boegel EPII Wet Time	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
2 minutes	1.18	0.02	0.05	0.07	0.14	0.18	0.20	94% co
4 minutes	1.08	0.06	0.07	0.08	0.18	0.22	0.25	95% co
6 minutes	1.10	0.06	0.08	0.12	0.19	0.22	0.23	95% co
8 minutes	1.09	0.04	0.06	0.08	0.16	0.22	0.22	94% co
10 minutes	1.09	0.04	0.06	0.10	0.16	0.22	0.23	95% co
12 minutes	1.09	0.05	0.07	0.10	0.16	0.23	0.23	95% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.3.2 Al 7075-T6 Boegel Wet Time Evaluation

Results of the wet time evaluation on Al 7075-T6 are shown in Table 5. As with Al 2024-T3, there appears to be no difference in either crack growths or failure modes of wedge test specimens when the wet time is varied between 2 minutes and 12 minutes. There is sufficient data to show that wet times from 2-20 minutes provide acceptable wedge test results on Al 7075-T6 grit-blasted surfaces since wet times of 20 minutes were evaluated in the grit-blast designed experiment. This experiment also shows there is little apparent difference between Al 2024-T3 and Al 7075-T6 alloys when grit-blasting, confirming both the alloy evaluation (3.1.2) and the grit-blast designed experiment (3.1.1).

Table 5: Effect of Boegel Wet Time on Al 7075-T6 Grit-Blast Wedge Test Results

Boegel EPII Wet Time	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
2 minutes	1.13	0.08	0.09	0.14	0.21	0.26	0.26	94% co
4 minutes	1.13	0.06	0.07	0.10	0.18	0.23	0.23	96% co
6 minutes	1.16	0.05	0.08	0.09	0.16	0.19	0.20	95% co
8 minutes	1.18	0.06	0.10	0.11	0.20	0.25	0.25	95% co
10 minutes	1.15	0.07	0.10	0.14	0.20	0.24	0.24	94% co
12 minutes	1.21	0.06	0.08	0.08	0.19	0.21	0.22	94% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.4 Boegel-EPII Drying Evaluation

3.1.4.1 Dry Method Evaluation

The grit-blast designed experiment (3.1.1) showed that the nitrogen force-dry method for drying Boegel-EPII provided better results in the wedge test than ambient drying at laboratory conditions. An experiment was conducted to determine the effect of force drying Boegel-EPII versus ambient drying for two different wet times, 3 minutes and 10 minutes.

Al 2024-T3 wedge test adherends composed of were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50 µm aluminum-oxide grit and blown with 35 psi compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for the specified times. Once the wet time had elapsed, panels were either force-dried with compressed nitrogen or placed in a vertical rack to dry for 60 minutes at ambient temperature (70°F). Once dried, adherends were primed with BR 6747-1. Primed adherends were dried at ambient temperature for 30 minutes and cured for 60 minutes at 250°F. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 140°F and 95-100% RH. Results are shown in Table 6. The different dry methods do not appear to affect the wedge test results for either the 3-minute wet time or the 10-minute wet time. Comparable results are obtained using either the ambient dry or force dry.

Table 6: Effect of Dry Method on Al 2024- T3 Grit-Blast Wedge Test Results

Dry Method	Wet Time	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
			1 hr	8 hr	24 hr	7 days	21 days	28 days	
Ambient dry	3 minutes	1.14	0.01	0.04	0.10	0.14	0.19	0.19	94% co
	10 minutes	1.12	0.01	0.05	0.08	0.17	0.19	0.19	94% co
Nitrogen Force Dry	3 minutes	1.13	0.03	0.07	0.10	0.17	0.22	0.24	95% co
	10 minutes	1.10	0.02	0.08	0.09	0.18	0.21	0.23	93% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.4.2 Dry Time Evaluation

The dry time evaluation was conducted in order to generate data for cases where a complete ambient dry of the Boegel-EPII solution (as opposed to force-drying) is desired or required. Two dry times were evaluated in the grit-blast designed experiment (section 3.1.1), 1 hour and 4 hours. No difference was detected in the designed experiment between the 1 and 4 hour dry times, so the intent of this additional experiment was to determine the minimal dry time required to achieve cohesive failure modes in the wedge test.

Wedge test adherends composed of Al 2024-T3 were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50μm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Adherends were placed in a vertical rack to dry for the given dry time at ambient temperature (70°F). Once the dry dime elapsed, adherends were primed with BR 6747-1. Primed adherends were dried at ambient temperature (70°F) for 30 minutes and cured for 60 minutes at 250°F. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 140°F and 95-100% RH. Results are shown in Table 7. The different evaluated dry times appear to have no effect on the crack growth or failure modes of grit-blasted wedge test specimens. It should be noted that the effect of varying drying temperature and humidity were not evaluated in this study. All drying was conducted at ambient laboratory conditions (70°F and 60% RH). Future work is required to evaluate the effects of temperature and humidity.

Table 7: Effect of Boegel Dry Time on Al 2024-T3 Grit-Blast Wedge Test Results

Ambient Dry Time	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
Force Dry w/N_2	1.17	0.01	0.04	0.07	0.12	0.14	0.16	95% co
15 minutes	1.14	0.02	0.05	0.09	0.14	0.21	0.22	95% co
30 minutes	1.18	0.01	0.06	0.09	0.15	0.21	0.22	96% co
45 minutes	1.13	0.04	0.07	0.09	0.13	0.20	0.20	95% co
60 minutes	1.15	0.01	0.03	0.08	0.12	0.15	0.17	95% co
75 minutes	1.14	0.04	0.07	0.09	0.14	0.19	0.19	94% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.5 Primer Evaluation

Adhesive bond primers provide a number of benefits for adhesive joints[11]. The use of bond primers allows treated, unbonded panels to be stored for long periods of time prior to adhesive bonding without repeating the surface preparation. Bond primers may significantly increase bond environmental durability. Although a need does not exist for storing treated aluminum for long periods prior to repair bonding, the effect of bond primers on bond durability is a concern. This section provides data comparing different primers and varying primer cure cycles.

3.1.5.1 Effect of Primer Type

In the grit-blast designed experiment (section 3.1.1), wedge test specimens primed with Cytec BR 6747-1 were compared to wedge tests specimens fabricated without bond primer. Results of that experiment showed that BR 6747-1 increased the bond durability in the wedge test when compared to specimens without primer. In this experiment, wedge test specimens were fabricated with the following bond primers:

- ***Cytec BR 127:*** solvent-based, chromated bond primer,
- ***Cytec BR 6747-1:*** waterborne, chromated bond primer,
- ***Cytec BR 6757-1:*** waterborne, nonchromated bond primer, and
- ***No bond primer.***

Al 2024-T3 wedge test adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50µm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Adherends were force dried with compressed nitrogen and primed (if required). Primed adherends were dried at ambient temperature (70°F) for 30 minutes and cured for 60 minutes at 250°F. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at both 120°F and 95-100% RH as well as 140°F and 95-100% RH. Results of the wedge tests performed at 120°F and 95-100% RH is shown in Table 8. Results of the wedge tests performed at 140°F and 95-100% RH are shown in Table 9.

Table 8: Effect of Primer on Grit-Blast Wedge Test Results at 120°F and 95-100% RH

Bond Primer	Initial (in)	Cummulative Crack Growth (in)						Failure Mode
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
BR 127	1.16	0.09	0.13	0.15	0.20	0.26	0.29	7% co
BR 6747-1	1.17	0.03	0.05	0.09	0.09	0.15	0.15	97% co
BR 6757-1	1.13	0.05	0.05	0.06	0.10	0.12	0.12	68% co*
No primer	1.09	0.08	0.12	0.15	0.20	0.22	0.23	84% co**

co: cohesive failure

* non-cohesive failure occurred at the primer-adhesive interface

** non-cohesive failure occurred between the aluminum and adhesive

Table 9: Effect of Primer on Grit-Blast Wedge Test Results at 140°F and 95-100% RH

Bond Primer	Initial (in)	Cummulative Crack Growth (in)						Failure Mode
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
BR 127	1.19	0.08	0.16	0.19	0.27	0.33	0.33	0% co
BR 6747-1	1.06	0.03	0.11	0.11	0.15	0.23	0.24	95% co
BR 6757-1	1.16	0.03	0.07	0.11	0.17	0.17	0.20	83% co*
No primer	1.22	0.07	0.11	0.19	0.26	0.28	0.33	36% co**

co: cohesive failure

* non-cohesive failure occurred at the primer-adhesive interface

** non-cohesive failure occurred between the aluminum and adhesive

When testing at both 120°F and 140°F, only adherends primed with BR 6747-1 exhibit acceptable durability results. Specimens primed with BR 127 exhibited complete interfacial failure between the aluminum and primer. A compatibility problem may exist between the BR 127 bond primer and the Boegel-EPII coating, however the exact cause for failure is unknown at this time. BR 6757-1 exhibits interfacial failure that visually appears to occur between the primer and adhesive. It was difficult to use traditional methods such as energy-dispersive spectrometry (EDS) to determine the exact location of failure due to the fact BR 6757-1 does not contain chromium. In order to identify failure location, EDS was used to identify the location of chromium. Chromium was found in the BR 6747-1 bond primer but not the adhesive, so locations where chromium was detected contained primer.

3.1.5.2 Effect of Primer Cure

The requirement for separate primer and adhesive cure cycles creates undesirable time constraints for personnel performing repair adhesive bonding processes. Therefore, the ability to cocure the primer and adhesive in a single cure cycle was evaluated. The following three primer cure cycles were evaluated using BR 6747-1 since this primer showed the best performance in the primer type evaluation (section 3.1.5.1):

1. ***Precure (control):*** 30 minute dry at ambient temperature (70°F) and 60 minutes at 250°F according to the manufacturer’s recommendations prior to bonding,
2. ***Primer Fuse:*** 30 minute dry at ambient temperature followed by heat application via heat gun or oven to “fuse” the primer, then cocure with adhesive for 60 minutes at 250°F, and

3. ***Cocure:*** 30 minute dry at ambient temperature followed by adhesive application and cocure with the adhesive for 60 minutes at 250°F.

BR 6747-1 primer dries as a powdery film when applied. The dried surface appears to be rough and uneven. When cured, the film appears to be uniform and translucent. This same appearance is also achieved by adding heat via an oven or heat gun for a short period of time to flow the primer. This was called “fusing” the primer. Fusing the primer allows for easier handling of the panels since the dry powder could be damaged. Fusing does not cure the primer and would not necessarily result in the same performance obtained by completely curing the primer prior to bonding. When cocuring, adhesive was applied directly to the powdery surface so the primer and adhesive were cocured in a single cure cycle without first fusing the primer.

Al 2024-T3 wedge test adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was viably present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50μm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Adherends were force dried with compressed nitrogen and primed with BR 6747-1 which was then dried at ambient temperature for 30 minutes. Adherends were precured, fused, or cocured with the adhesive. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 140°F and 95-100% RH. Results are shown in Table 10. All three of the primer cure cycles provided similar crack growths and failure modes after 28 days of environmental aging.

Table 10: Effect of BR 6747-1 Primer Cure on Grit-Blast Wedge Test Results

Primer Cure Cycle	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
Precure	1.06	0.03	0.11	0.11	0.15	0.23	0.24	95% co
Primer fuse	1.06	0.02	0.14	0.14	0.18	0.25	0.28	93% co
Cocure	1.09	0.01	0.14	0.14	0.15	0.24	0.27	95% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.5.3 *Effect of Cocuring Primer and Adhesive Under Vacuum Pressure*

Cocuring the primer and adhesive in a single heat cycle saved processing time and did not appear to present any adverse effects when cured under positive pressure. However, most field-level bonded repairs are performed using vacuum to apply pressure. In order to determine the effect of cocuring the primer and adhesive under vacuum, wedge test panels were fabricated using a grit-blast deoxidation step.

Al 2024-T3 wedge test adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50μm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Adherends were dried at ambient temperature (70°F) for 30 minutes and primed with BR 6747-1. Primed adherends were dried at ambient temperature for 30 minutes. Adherends for one wedge test panel were precured according to the manufacturer's recommendations and bonded using 35-psi positive pressure during cure. Adherends for another wedge test panel were fabricated using primer cocured with the adhesive under 27 inches Hg vacuum. Both wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F in a portable autoclave utilizing the different cure pressures. Panels were machined into specimens, measured for bondline thickness, and tested at 120°F and 95-100% RH. Results are shown in Table 11. There did not appear to be any difference in crack growth or failure mode due to cocuring under vacuum pressure.

Table 11: Effect of Cocuring Primer and Adhesive Using Vacuum Pressure on Grit-Blast Wedge Test Results

Primer Cure Cycle	Adhesive Cure Pressure	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
			1 hr	8 hr	24 hr	7 days	21 days	28 days	
Precure	35 psi	1.17	0.03	0.05	0.05	0.09	0.15	0.15	97% co
Cocure	27 in Hg	1.12	0.04	0.05	0.06	0.10	0.13	0.14	98% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.5.4 Effect of Primer Application Method

Field-level and depot maintenance personnel have expressed interest in the ability to apply bond primers without the use of a spray gun. It is easier to apply primer with a brush or cloth versus applying primer with a spray gun and health and safety concerns are increased when hazardous materials such as chromium are atomized in the air, especially in poorly ventilated areas. For these reasons, an experiment was conducted to determine the effect of applying primer with a lint-free cloth versus spray-application.

Two wedge test panels per condition were fabricated for this experiment. Al 2024-T3 wedge test adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50μm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Adherends were force dried with compressed nitrogen and primed with BR 6747-1 using both a Binks 105 spray gun and a lint-free cloth. Primed adherends were dried at ambient temperature (70°F) for 30 minutes and cocured with the adhesive. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 120°F and 95-100% RH. Results are shown in Table 12. Although there was little difference in amount of crack growth due to the primer application method, there was a difference in failure mode. The specimens primed with a lint-free cloth exhibited lower percentages of cohesive failure than the panels primed with the spray gun. Future work will be conducted due to the need for nonspray priming techniques.

Table 12: Effect of Primer Application Method on Grit-Blast Wedge Test Results

Primer Application Method	**Initial (in)**	**Cummulative Crack Growth (in)**							**Failure Mode***
		1 hr	**8 hr**	**24 hr**	**7 days**	**14 days**	**21 days**	**28 days**	
Binks 105 spray gun	1.16	0.04	0.07	0.07	0.14	0.15	0.16	0.18	98% co
	1.10	0.04	0.05	0.08	0.11	0.13	0.14	0.17	97% co
Lint-free cloth	1.05	0.07	0.07	0.11	0.15	0.17	0.17	0.20	91% co
	1.19	0.05	0.07	0.11	0.15	0.17	0.18	0.19	91% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.6 Effect of Epoxy Film Adhesive

Although 3M Company AF 163-2M adhesive was chosen for use in optimizing the sol-gel surface preparations, a number of other 250°F-curing epoxy-film adhesives are used for field and depot-level bonded repair. Therefore, wedge test panels were bonded with 3M Company AF 163-2M (control), Cytec FM 73M, Loctite Hysol EA 9628, and Loctite Hysol EA 9696 adhesives to determine the effect of different film adhesives. Each adhesive was 0.06 psf weight and was manufactured with a mat carrier.

Al 7075-T6 wedge test adherends were cleaned with acetone-soaked lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50μm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Adherends were force dried with compressed nitrogen and primed with BR 6747-1. Primed adherends were dried at ambient temperature (70°F) for 30 minutes and cured according to the manufacturer's recommendations. Wedge test panels were bonded with adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 140°F & 95-100% RH. Results are shown in Table 13. AF 163-2M specimens exhibited the largest crack growth and largest nicks of adhesive failure mode at the edges of the specimens after 28 days in humidity. Specimens bonded with FM 73M, EA 9628, and EA 9696 exhibited cohesive failure modes. Cytec FM 73M, Loctite Hysol EA 9628 and Loctite Hysol EA 9696 all appear to yield acceptable wedge test results when used with the grit-blast deoxidation step and Boegel-EPII solution.

Table 13: Effect of Adhesive Type on Grit-Blast Wedge Test Results

Adhesive	Initial (in)	Cummulative Crack Growth (in)					Failure Mode*
		1 hr	8 hr	24 hr	7 days	28 days	
3M Company AF 163-2M	1.15	0.05	0.08	0.13	0.18	0.26	94% co
Cytec Fiberite FM 73M	1.26	0.00	0.01	0.01	0.06	0.12	96% co
Loctite Hysol EA 9628	1.36	0.00	0.00	0.03	0.08	0.16	100% co
Loctite Hysol EA 9696	1.29	0.02	0.04	0.05	0.12	0.17	100% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.1.7 Initial Bond Strength Results

In order to determine the initial strengths of bonded joints using Boegel-EPII over a grit-blasted surface, tensile lap shear[12] and floating roller peel[13] tests were conducted. This was to ensure the initial strength of grit-blasted bonded joints treated with Boegel-EPII, without the effect of moisture conditioning, is similar to that of PAA-prepared bonded joints.

Al 2024-T3 adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with general purpose 3M Company Scotch-Brite™ nylon pads until a shiny baseline surface was obtained. Adherends were then blasted with 50µm aluminum-oxide grit and blown with compressed nitrogen to remove as much residual grit from the surface as possible. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Adherends were force dried with compressed nitrogen. Primed specimens were primed with BR 6747-1 and dried at ambient temperature (70°F) for 30 minutes. Some specimens were precured according to the manufacturer's recommendations and others were cocured with the adhesive (without fusing). All panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Lap shear testing was performed at –65°F, 70°F, and 180°F after a four-minute soak at temperature. Floating roller peel testing was performed at 70°F and –65°F after a four-minute soak at temperature. Published data from 3M Company[14] on AF 163-2M using chromic acid anodize (CAA) primed with EC-3917 as well as PAA control panels primed with BR 6747-1 and cured according to the manufacturer's recommendations were used to compare initial strength results. Lap shear results are shown in Table 14. Floating roller peel test results are shown in Table 15. All data points are the average of five specimens.

Table 14: Tensile Lap Shear Test Results for Grit-Blasted Al 2024-T3

Surface Prep	Primer	Lap Shear Strength (psi) [% Cohesive Failure]		
		-65°F	**70°F**	**180°F**
grit-blast sol-gel	BR 6747-1 [p]*	5778 [68%]	5796 [100%]	3770 [22%]
grit-blast sol-gel	BR 6747-1 [c]**	5946 [84%]	5382 [100%]	3644.1 [77%]
grit-blast sol-gel	none	6038 [91%]	5430 [98%]	3773 [22%]
PAA	BR 6747-1 [p]	6398 [56%]	6028 [100%]	4159 [28%]
Published data on CAA Al 2024-T3	EC-3917	6400	5700	3600

* [p]: primer precured according to manufacturer's recommendations

** [c]: primer cocured with adhesive

Table 15: Floating Roller Peel Test Results for Grit-Blasted Al 2024-T3

Surface Prep	Primer	Peel Strength (pli) [% cohesive]	
		-65°F	70°F
grit-blast sol-gel	BR 6747-1 [p]*	51.1 [43%]	76.6 [100%]
grit-blast sol-gel	BR 6747-1 [c]**	64.3 [79%]	68.8 [100%]
PAA	BR 6747-1 [p]*	62.2 [90%]	72.7 [100%]
Published data on FPL etched Al 2024-T3	EC-3924B	58.0	79.0

* [p]: primer precured according to manufacturer's recommendations

** [c]: primer cocured with adhesive

3.2 Optimization of Processing Parameters Using Boegel-EPII with Nylon Pad Abrasion Surface Activation Techniques

As anticipated (Section 3.1), the use of grit-blasting with Boegel-EPII solution produced an excellent surface for adhesive bonding with good initial strength and bond durability results. However, one goal of this project is to eliminate grit-blasting since it can be difficult to properly perform, particularly on aircraft. Containment and clean-up efforts represent a significant inconvenience and increased repair time, especially for bonding applications in sensitive areas of the aircraft such as inside wing fuel tanks.

The use of nylon pads to abrade the surface prior to application of the Boegel-EPII solution would be a simple replacement for the grit-blasting step. It was anticipated that the more reactive Boegel EPII chemistry might yield acceptable moisture durability results as measured by the wedge test, whereas this approach was not successful for the silane surface preparation[15]. Abrasion could be accomplished using an air-driven rotary tool that is available in most field-level maintenance facilities. However, a potential drawback to nylon pad-abrasion is the increased difficulty in visually detecting organic contamination on the abraded surface.

In order to optimize the process parameters involved with activating the bonding surface and subsequent application of Boegel-EPII and bond primers, a designed experiment was conducted to determine significant processing factors. Once identified, several smaller experiments were conducted to determine optimum operating windows for individual steps in the surface preparation.

3.2.1 Nylon Pad Abrasion Designed Experiment

In order to evaluate several key processing factors using a nylon pad-abraded deoxidation process with Boegel-EPII solution, a designed experiment was conducted using an L16 array. The evaluated processing factors are listed in Table 16. Aluminum alloy type, grind time, time between deoxidation and application of Boegel-EPII solution (post-abrade time), Boegel-EPII wet time, Boegel-EPII dry method, Boegel-EPII dry time, and bond primer cure cycle factors were all evaluated using a matrix consisting of 16 wedge test panels.

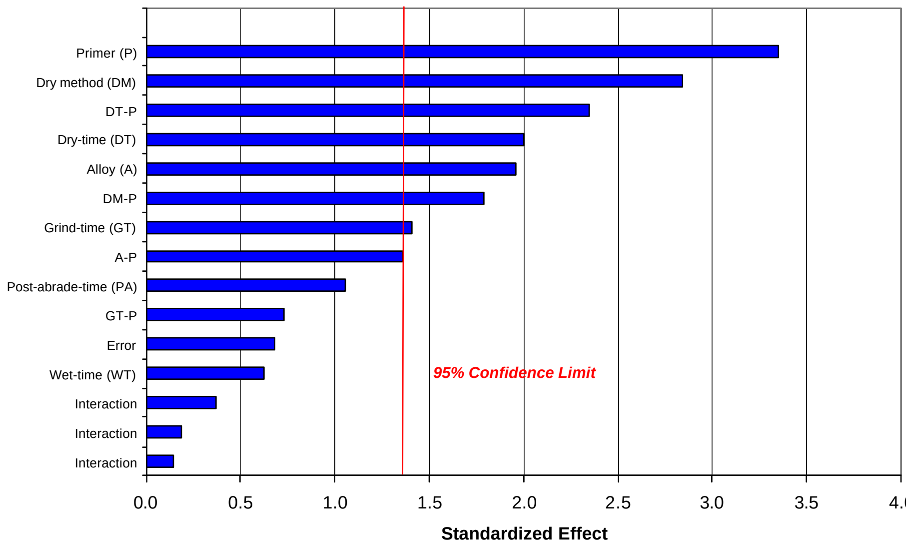
Table 16: Nylon Pad Deoxidation Designed Experiment Processing Parameters

Factor	Parameter #1	Parameter #2
Alloy	Al 2024-T3	Al 7075-T6
Grind time	1 minute	2 minutes
Post-abrade time	<1 minute	30 minutes
Boegel-EPII wet time	3 minutes	10 minutes
Dry method	nitrogen force	ambient dry
Dry time	30 minutes	60 minutes
Primer cure	Precure	Cocure

Wedge test panels were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination were visibly present. The panels were then abraded with general purpose Scotch-Brite™ pads until a polished surface was obtained. This initial nylon pad abrasion step was used to generate a baseline surface to start the process. All panels were then abraded with 3-inch diameter Standard Abrasive fine "Buff and Blend" pads on a 20,000-RPM high-speed grinder for the specified grind time. Clean, dry compressed nitrogen was used to drive the high-speed grinder in order to prevent contamination from oil, condensed moisture, or other contaminants. After the panels were abraded, 40 psi-compressed nitrogen was used to remove as much residue from the surface as possible. At this point, panels were allowed to sit for a specified time (post-abrade time) at ambient conditions (70°F and 60% relative humidity) to determine if a minimum time requirement existed between deoxidation and Boegel-EPII solution application. Boegel-EPII solution was applied via brush within 1 minute or after 30 minutes of abrasion, and the surface was kept wet for the specified time. The panels were ambient dried or force-dried using 40 psi nitrogen. Once dry, panels were primed with BR 6747-1 primer to a thickness of 0.1-0.3 mil. Primed panels were dried at ambient conditions for 30 minutes prior to cure. The primer cure was accomplished in one of two ways: (1) precuring at 250°F for 60 minutes per manufacturer's directions after drying for 30 minutes at 70°F, or (2) drying for 30 minutes at ambient temperature then "fusing" the primer using a heat gun followed by cocuring with AF 163-2M adhesive for 60 minutes at 250°F.

Panels were machined into 1.0-inch wide specimens. The bondline of each specimen was measured with an optical microscope. The specimens were then tested at 120°F and 95-100% RH. The crack growths of the specimens after 28 days were used to calculate the significance of each factor using the design of experiments philosophy. Figure 9 shows the calculated significance of each factor to the 95% confidence limit. All factors or interactions having a standardized effect greater than the 95% confidence limit were considered significant.

Figure 9: Significance of Nylon Pad Deoxidation Processing Factors



The wet time and post-abrade time factors, along with a few interactions, were considered insignificant. The optimum set of processing factors for the nylon pad/sol-gel surface preparation is listed in Table 17. The two most significant factors are dry method and primer cure method. Panels that were ambient dried and cocured with the adhesive performed the best.

Table 17: Optimum Processing Parameters for Nylon Pad Deoxidation Process

Factor	Optimum Parameter
Alloy	Al 2024-T3
Grind time	2 minutes
Dry method	ambient dry
Dry time	30 minutes
Primer cure	Cocure
*Post-abrade time**	*<1 minute*
*Boegel-EPII wet time**	*10 minutes*

* insignificant

Since there were no panels fabricated with the complete set of "optimal" processing parameters as determined by the designed experiment, a separate experiment was conducted to validate the experiment. Al 2024-T3 wedge test specimens were fabricated with AF 163-2M utilizing the designed experiment optimum nylon pad process. The specimens were tested at 120°F and 95-

100% RH as well as 140°F and 95-100% RH. Results are shown in Table 18. Cohesive failure modes were witnessed after 28 days at 120°F and 95-100% RH (Figure 10). However, a small amount of adhesive failure occurred at the edges and toward the center of the AF 163-2M specimens that were tested at 140°F and 95-100% RH (Figure 11). The verification panels validated the designed experiment since all specimens tested at 120°F failed cohesively after 28 days of exposure.

Table 18: Nylon Pad Designed Experiment Verification

Testing Conditions	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
120°F & 95-100% RH	1.13	0.00	0.08	0.12	0.13	0.13	0.14	96%co
140°F & 95-100% RH	1.19	0.04	0.09	0.09	0.19	0.26	0.27	91% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Figure 10: Failure Mode Exhibited by "Optimal" Nylon Pad Deoxidized Specimens Treated with Boegel-EPII and Tested at 120°F and 95-100% Relative Humidity

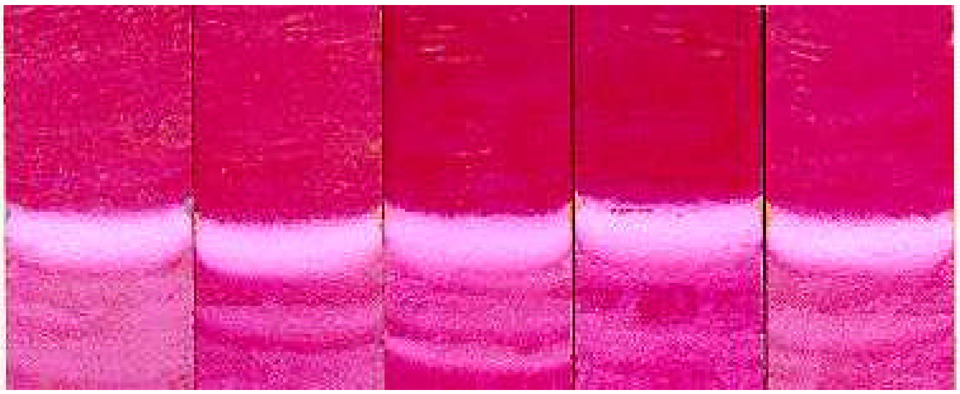


Figure 11: Failure Mode Exhibited by "Optimal" Nylon Pad Deoxidized Specimens Treated with Boegel-EPII and Tested at 140°F and 95-100% Relative Humidity

3.2.2 Effect of Solvent Type on Nylon Pad Deoxidation Process

Environmental regulations vary from location to location. Numerous locales have tight regulations restricting the use of volatile solvents such as methylethyl ketone (MEK). Due to the variability of these restrictions, several different solvents are used depending on the restrictions in place at the repair facilities. Therefore, wedge test panels were fabricated with various solvents used for degreasing in order to determine if the solvent caused differences in bond durability. Three different solvents were used in this investigation: (1) acetone, as used in the designed experiment (3.2.1), (2) MEK, and (3) isopropyl alcohol (IPA).

Wedge test adherends composed of Al 2024-T3 were cleaned with the given solvent and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company Scotch-Brite™ Roloc™ medium nylon pads for 90 seconds using a 20,000-RPM nitrogen-driven grinder. Adherends were wiped with a lint-free cloth soaked with the appropriate solvent to clean the surface prior to application of Boegel-EPII. Boegel-EPII solution was applied using an acid brush immediately upon completion of the solvent wipe, and the surfaces were kept wet for 3 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. Primed adherends were dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH. Results are shown in Table 19.

There appears to be no difference in either crack growth or failure mode due to solvent type. However it should be noted that the adherends used in this program were fairly clean to prior to processing as compared to typical aircraft structure that may be contaminated with dirty water, oil, fuel, or hydraulic fluid. Therefore, the full effect of using different solvents for degreasing, especially in a field level environment, was not accurately depicted in this experiment.

Table 19: Effect of Solvent Type on Nylon Pad Wedge Test Results

Solvent	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Isopropyl alcohol	1.11	0.04	0.07	0.09	0.13	0.15	0.17	0.18	96%co
Acetone	1.19	0.04	0.07	0.09	0.13	0.15	0.17	0.17	96%co
MEK	1.13	0.06	0.09	0.11	0.16	0.18	0.18	0.20	96% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.3 Effect of Nylon Pad Manufacturer

Several types of nylon pads are currently manufactured, sold, and used commercially. 3M Company and Standard Abrasives are among the current manufacturers. In order to determine the variability in the performance of the nylon pad due to manufacturer and pad grade (coarseness), an experiment was conducted using different types and grades of nylon pads for deoxidation prior to application of Boegel-EPII solution.

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with various types of 3-inch diameter nylon pads for 90 seconds using a 20,000-RPM nitrogen-driven grinder. Standard Abrasives 3-inch diameter "Buff and Blend" disks and 3M Company 3-inch diameter Roloc™ pads were used in this experiment. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. The primer was dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH. Results are shown in Table 20. Overall, panels abraded with the 3M Company pads appear to yield smaller crack growths and higher percentages of cohesive failure than the Standard Abrasives pads. The 3M medium and coarse pads and Standard Abrasive medium pads provided the best overall results in the wedge test.

Table 20: Effect of Nylon Pad Grade and Manufacturer on Nylon Pad Wedge Test Results

Nylon Pad	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
3M Very Fine	1.08	0.02	0.09	0.10	0.12	0.16	0.16	95% co
3M Medium	1.14	0.00	0.04	0.05	0.07	0.10	0.10	97% co
3M Coarse	1.09	0.02	0.07	0.08	0.09	0.11	0.12	98% co
SA Fine	1.07	0.04	0.09	0.10	0.15	0.19	0.19	94% co
SA Medium	1.09	0.02	0.09	0.10	0.13	0.15	0.15	97% co
SA Coarse	1.11	0.03	0.13	0.13	0.15	0.19	0.21	94% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.4 Effect of Abrasion Time

Quantifying a properly abraded surface for adhesive bonding is important for quality control. This is particularly difficult for a nylon pad-abraded surface where the process of abrading is heavily dependent on the technician performing the operation. In an attempt to determine the minimal amount of abrading required to effectively deoxidize an aluminum surface for adhesive bonding, several wedge test panels were fabricated while varying the amount of time used to abrade the bond surfaces. The evaluated abrading times (30 to 120 seconds) were practical for a 6.5 inch by 6.5 inch wedge adherend. Less than 30 seconds abrasion for a 6.5 inch by 6.5 inch area would not have been enough time to abrade the entire panel. More than 120 seconds per 6. inch by 6.5 inch area would not be practical on large parts in a field environment due to time limitations.

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company coarse Roloc™ pads for various times using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. The primer was dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F

& 95-100% RH. Results are shown in Table 21. There does not appear to be any correlation between abrasion time and wedge test results since all specimens failed cohesively with very similar crack growths.

Table 21: Effect of Abrasion Time on Nylon Pad Wedge Test Results

Abrasion Time	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
30 seconds	1.12	0.01	0.03	0.05	0.14	0.15	0.16	0.16	95% co
60 seconds	1.12	0.04	0.06	0.07	0.11	0.12	0.15	0.15	96% co
90 seconds	1.16	0.03	0.04	0.08	0.12	0.13	0.15	0.15	96% co
120 seconds	1.14	0.01	0.04	0.06	0.11	0.14	0.14	0.14	96% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.5 Effect of Time Between Deoxidation and Application of Boegel-EPII Solution

During the nylon pad designed experiment (3.2.1), the time between deoxidation and application of Boegel-EPII solution (post-abrade time) was evaluated and determined to be insignificant between one and thirty minutes. In order to further evaluate this processing factor and determine the operating window, a follow-on experiment was conducted to determine the effect of increased time between deoxidation using the nylon pad/sol-gel process.

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company medium Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3minutes. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. The primer was dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH. Results are shown in Table 22. Although the failure mode is lower for the 30-minute wait prior to application of Boegel-EPII solution, it does not appear that a drastic change in wedge test performance was detected due to time between deoxidation and application of Boegel-EPII solution. From the results of the designed experiment and this experiment, it appears the time between the deoxidation step and application of Boegel-EPII solution is insignificant for times between 1 and 120 minutes.

Table 22: Effect of Increased Time Between Deoxidation Using Nylon Pads and Application of Boegel-EPII Solution

Post-Deoxidation Time	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
within 1 minute	1.17	0.02	0.07	0.08	0.16	0.18	0.18	0.20	95% co
30 minutes	1.16	0.06	0.11	0.12	0.17	0.19	0.21	0.22	89% co
90 minutes	1.16	0.06	0.11	0.12	0.17	0.19	0.21	0.22	95% co
120 minutes	1.11	0.00	0.08	0.08	0.10	0.13	0.15	0.15	98% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.6 Boegel-EPII Wet Time Evaluation

During the nylon pad designed experiment (section 3.2.1), the effect of varying the wet time of Boegel-EPII solution on an abraded surface was evaluated for 3 minutes and 10 minutes. Although the wet time processing factor was found to be insignificant to the 95% confidence level, additional data was desired. Therefore, an experiment was conducted to determine the effect of varying wet time between 2 and 20 minutes.

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company medium Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for the given times. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. The primer was dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH.

Results are shown in Table 23. Wetting the nylon pad-abraded surface for times between 2 minutes and 20 minutes appeared to have no effect on wedge test results.

Table 23: Effect of Boegel-EPII Wet Time on Nylon Pad Wedge Test Results

Wet Time	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
2 minutes	1.17	0.07	0.11	0.11	0.16	0.18	0.18	0.18	99% co
4 minutes	1.13	0.08	0.13	0.13	0.17	0.18	0.20	0.24	97% co
6 minutes	1.12	0.08	0.10	0.10	0.16	0.18	0.18	0.20	99% co
8 minutes	1.15	0.03	0.10	0.11	0.17	0.18	0.18	0.19	99% co
10 minutes	1.14	0.06	0.08	0.09	0.12	0.15	0.18	0.18	99% co
12 minutes	1.13	0.04	0.07	0.08	0.12	0.13	0.14	0.16	99% co
20 minutes	1.13	0.05	0.10	0.11	0.15	0.17	0.18	0.19	98% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.7 Effect of Boegel-EPII Dry Method

Upon completion of the Boegel-EPII wet time, the bonding surface must be dried before application of bond primer or adhesive. Two methods were evaluated during the nylon pad designed experiment (section 3.2.1), a force dry with 40-psi compressed nitrogen and a 30-minute ambient-temperature dry cycle in a controlled laboratory environment. The designed experiment showed that panels dried vertically for 30 minutes at ambient temperature exhibited better results than panels blown dry with 40-psi compressed nitrogen. However, when performing surface preparations in the field or at depot level, maintenance personnel do not want to wait 30 minutes to begin the next processing step, and there is also a danger of contaminating the bonding surface during this 30-minute drying period. In addition, the surfaces to be treated may exist at an orientation other than vertical, and may possess geometry that allows the sol-gel solution to puddle and not readily dry in 30 minutes and/or result in excessively thick sol-gel film. Therefore, force drying with nitrogen or clean, dry air would be more practical. In order to determine the effect of Boegel-EPII solution drying method, an experiment was conducted varying the drying method (ambient and force-dry), ambient dry time (10 minutes and 30 minutes), and nitrogen line pressure (5 to 50 psi).

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company medium Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for the given times. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried either at ambient laboratory temperature or force dried with compressed nitrogen. Various dry times and nitrogen pressures were used to dry the adherends. Once dried, panels were primed with BR 6747-1, dried at ambient conditions (70°F and 60% RH) for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH. Results are shown in Table 24.

When ambient-drying in a controlled laboratory environment, there appears to be no difference between drying for 30 minutes or 10 minutes prior to application of the primer. However, drying times in the field or depot will likely be different due to the actual temperature and humidity experienced while performing the surface preparation. When force-drying the panels using compressed nitrogen, good failure modes are noticed up to pressures of 40 psi with a stand-off distance of 6-8 inches. A loss in percentage of cohesive failure mode is noticed when drying panels with 50-psi nitrogen. Panels dried with 5-20 psi nitrogen exhibited crack growth and failure modes similar to those of panels dried at ambient laboratory conditions for 30 minutes. Stand-off distance was not evaluated as a parameter of drying in this experiment although it could be a significant factor. This could be especially true if the stand off distance were too short, causing higher pressures on the bond surfaces.

Table 24: Effect of Drying Method on Nylon Pad Wedge Test Results

Dry Method	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
30 min @ RT	1.10	0.05	0.09	0.13	0.16	0.17	0.20	0.21	98% co
10 min @ RT	1.12	0.02	0.08	0.11	0.18	0.20	0.20	0.23	97% co
N_2 force dry (5 psi)	1.11	0.01	0.01	0.03	0.10	0.12	0.14	0.18	99% co
N_2 force dry (10 psi)	1.12	0.02	0.03	0.05	0.11	0.13	0.15	0.20	98% co
N_2 force dry (20 psi)	1.15	0.00	0.05	0.08	0.15	0.16	0.17	0.22	99% co
N_2 force dry (30 psi)	1.20	0.02	0.05	0.10	0.14	0.15	0.17	0.22	94% co
N_2 force dry (40 psi)	1.10	0.03	0.05	0.08	0.13	0.15	0.15	0.20	99% co
N_2 force dry (50 psi)	1.21	0.07	0.11	0.13	0.19	0.23	0.26	0.31	80% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.8 Effect of Primer Application Method

Although spray application of bond primer in a laboratory setting is convenient, priming in field-level and depot environments with spray guns is often difficult due to equipment limitations and safety regulations concerning hazardous airborne materials. Therefore, maintenance personnel would like the option of applying bond primer using a manual-wipe method. This could include brushing, rolling, or wiping with some type of cloth. For this evaluation, wiping the primer with a dust-free cloth was compared to applying the primer with a spray gun, analogous to the evaluation conducted for grit-blast/sol-gel (section 3.1.5.4).

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company medium Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes. Once dried, panels were primed with BR 6747-1 either by spray application or by wiping primer on panels using a lint-free cloth. Primer had to be applied in a single step when wiping because secondary wipes actually removed primer solids. Once dried at ambient temperature for 30 minutes, adhesive was applied and the primer was cocured with the 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH. Two wedge crack extension test panels were fabricated for each condition. Results are shown in Table 25. Using a spray gun to apply the primer resulted in consistent cohesive failure modes. However, using a lint-free cloth to apply the primer resulted in one panel resulting in roughly 97% cohesive failure and the other with 93% cohesive failure. It should be noted that no effort was made to optimize the nonspray application technique. This will be undertaken in a future effort.

Table 25: Effect of Primer Application Method on Nylon Pad Wedge Test Results

Primer Application Method	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Spray Gun Application	1.10	0.07	0.10	0.11	0.18	0.18	0.21	0.23	97% co
	1.14	0.03	0.08	0.08	0.01	0.16	0.19	0.19	98% co
Wipe Application Using Lint-Free Cloth	1.12	0.09	0.11	0.13	0.15	0.17	0.19	0.20	93% co
	1.17	0.07	0.07	0.11	0.13	0.15	0.17	0.18	97% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.9 Primer Cure Evaluation

The effect of altering the cure cycle for BR 6747-1 primer from the manufacturer's recommended cure (precure) was evaluated using a grit-blast activation step in section 3.1.5.2. Primer fuse and cocure processes were two alternate cure cycles evaluated to decrease the amount of time required to perform a bonded repair. Precure and cocure processes were evaluated using the nylon pad activation step in the nylon pad designed experiment (3.2.1). However, more data was desired to establish the baseline properties when curing the primer under different conditions. Three primer cure cycles were evaluated using BR 6747-1:

1. ***Precure (control):*** 30 minute dry at ambient temperature (70°F) and 60 minutes at 250°F according to the manufacturer's recommendations prior to bonding,
2. ***Primer Fuse:*** 30 minute dry at ambient temperature followed by heat application via heat gun or oven to "fuse" primer then cocure with adhesive for 60 minutes at 250°F, and
3. ***Cocure:*** 30 minute dry at ambient temperature followed by adhesive

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company medium Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes. Once dried, panels were primed with

BR 6747-1 using a spray gun. Adherends were precured, fused, or cocured with the adhesive. Wedge test panels were bonded with AF 163-2M adhesive and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness, and tested at 120°F and 95-100% RH. Results are shown in Table 26. A small reduction in the amount of cohesive failure was detected in the "primer fuse" specimens.

Table 26: Effect of Primer Cure on Nylon Pad Wedge Test Results

Primer Cure Cycle	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Precure	1.11	0.08	0.10	0.12	0.14	0.18	0.19	0.19	96% co
Primer fuse	1.10	0.06	0.10	0.11	0.17	0.20	0.23	0.23	91% co
Cocure	1.14	0.09	0.11	0.15	0.18	0.19	0.20	0.20	98% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.10 Effect of Aluminum Alloy

Several different aluminum alloys are used for the manufacture of aircraft components. Since adhesive bonds will be used for a variety of alloys, most 2000 or 7000 series aluminum, an experiment was conducted to determine the effect of bonding to either Al 2024-T3 or Al 7075-T6, analogous to the evaluation conducted for grit-blast/sol-gel (section 3.1.2).

Wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company medium Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. Primed panels were dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH. Results are shown in Table 27. There appears to be no difference in the failure mode or crack growths due to aluminum alloys tested.

Table 27: Effect of Aluminum Alloy on Nylon Pad Wedge Test Results

Aluminum Alloy	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
Al 2024-T3	1.06	0.02	0.07	0.10	0.10	0.15	0.15	94% co
Al 7075-T6	1.16	0.02	0.04	0.07	0.09	0.15	0.15	95% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.11 Effect of Cure Cycle Pressure Application Technique

In order to achieve the best mechanical properties from an adhesive, it must typically be processed with positive pressure in an autoclave. However, when performing a bonded repair at the depot or in the field, particularly on aircraft, applying positive pressure is difficult. Therefore, it is common practice to use a vacuum bag to apply pressure to a bonded repair. However, this approach can increase porosity content and lead to weaker bond strength[16]. Therefore, wedge tests were conducted in order to determine the effect of cure pressure on bond durability. Tensile lap shear and floating roller peel tests were conducted using vacuum cure cycles to evaluate the effect on strength. Those data can be found in section 3.2.14.

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company coarse Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. Primed panels were dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F using 35-40 psi positive pressure, 15 inches Hg vacuum pressure or full vacuum pressure (27 inches Hg). Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F & 95-100% RH. Results are shown in Table 28. Although the vacuum-cured specimens exhibited shorter crack growths, the failure modes of the positive pressure and vacuum specimens were all cohesive. Therefore, it did not appear as if vacuum curing altered the durability of the adhesive bond.

Table 28: Effect of Cure Pressure on Nylon Pad Wedge Test Results

Cure Pressure	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
27 inches Hg	1.12	0.04	0.05	0.06	0.10	0.13	0.13	0.14	98% co
15 inches Hg	1.20	0.04	0.08	0.10	0.14	0.17	0.17	0.17	98% co
35-40 psi	1.10	0.05	0.09	0.13	0.16	0.17	0.20	0.21	98% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.12 Effect of Epoxy Film Adhesive

Although 3M Company AF 163-2M adhesive was chosen for use in optimizing the sol-gel surface preparations, a number of other 250°F-curing epoxy-film adhesives are used for field and depot-level bonded repair. Therefore wedge test panels were bonded with 3M Company AF 163-2M (control), Cytec FM 73, and Hysol EA 9628 adhesives to determine the effect of different film adhesives. Each adhesive was 0.06 psf weight. AF 163-2M and EA 9628 and was manufactured with a mat carrier while FM 73 was manufactured with a knit carrier. Testing in this section is analogous to the evaluation conducted for grit-blast/sol-gel (section 3.1.6).

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter Standard Abrasives fine pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 10 minutes. Abraded panels were coated with Boegel-EPII solution within 1 minute of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. Primed panels were dried at ambient temperature for 30 minutes and cocured with adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness, and tested at both 120°F & 95-100% RH and 140°F & 95-100% RH. Results of the specimens tested at 120°F & 95-100% RH are shown in Table 29. Results of the specimens tested at 140°F & 95-100% RH are shown in Table 30.

When tested at 120°F and 95-100% RH, specimens bonded with the three adhesives exhibit good crack growth and failure modes. However, when tested at 140°F and 95-100% RH, only specimens bonded with EA 9628 exhibit failure modes in excess of 95% cohesive. Similar specimens bonded with AF 163-2M and FM 73 exhibited lower percentages of cohesive failure.

Table 29: Effect of Film Adhesive on Nylon Pad Wedge Test Results when Tested at 120°F and 95-100% RH

Adhesive	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
AF 163-2M	1.07	0.02	0.08	0.12	0.16	0.16	0.20	96% co
EA 9628	1.35	0.02	0.08	0.08	0.12	0.12	0.12	97% co
FM 73	1.06	0.00	0.02	0.05	0.07	0.08	0.08	94% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Table 30: Effect of Film Adhesive on Nylon Pad Wedge Test Results when Tested at 140°F and 95-100% RH

Adhesive	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
AF 163-2M	1.08	0.06	0.10	0.13	0.21	0.24	0.25	90% co
EA 9628	1.37	0.02	0.06	0.09	0.16	0.21	0.22	96% co
FM 73	1.00	0.05	0.10	0.12	0.20	0.23	0.25	90% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Although the FM 73 data appeared to be comparable to AF 163-2M data (Table 29 and Table 30), further work was conducted since personnel from WR/ALC-TIEDD (Robins AFB, GA) were unable to reproduce similar results using the same process. Wedge test panels were fabricated using Al 2024-T3 adherends and the same surface preparation utilized in the previous adhesive evaluation, however, the effect of precuring the BR 6747-1 primer was also evaluated. When required, primer was precured as described in section 3.2.9. Panels were bonded with FM 73 0.085 psf knit carrier adhesive and AF 163-2M 0.06 psf mat carrier adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness and tested at 120°F & 95-100% RH. Results are shown in Table 31. Cocured FM 73 wedge test specimens exhibited a larger amount of adhesive failure when compared to cocured AF 163-2M specimens. This phenomenon has been repeated in follow-on testing by both AFRL/MLSA and WR/ALC-TIEDD. The reason for this phenomenon is unknown at this time.

Table 31: Additional FM 73 Wedge Test Data at 120°F and 95-100% RH

Primer Cure	Adhesive	Initial (in)	Cummulative Crack Growth (in)							Failure Mode
			1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Cocure	AF 163-2M	1.05	0.07	0.10	0.14	0.18	0.18	0.20	0.20	99% co
	FM 73	1.03	0.07	0.12	0.14	0.15	0.17	0.21	0.22	87% co
Precure	AF 163-2M	1.07	0.01	0.07	0.11	0.15	0.16	0.19	0.19	99% co
	FM 73	1.03	0.04	0.07	0.12	0.13	0.14	0.16	0.17	99% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.13 Effect of Testing Conditions on Wedge Test Results

Results in the wedge test can vary greatly depending on the aging conditions. The testing condition used during the grit-blast deoxidation experiments was 140°F and 95-100% RH. However, early work using the nylon pad deoxidation process revealed that testing at 140°F and 95-100% RH was too severe to show differences in processing steps. For that reason, a lower temperature testing condition of 120°F and 95-100% RH was used to detect differences in processing steps. This testing condition was used to evaluate the Australian silane surface preparation and a similar grit-blast/silane (GBS) surface preparation in the early 1990s[17]. Due to the success of fielded bonded joints prepared using GBS optimized via wedge tests at 120°F and 95-100% RH, these same testing conditions were used for work with the nylon pad deoxidation process. However, in order to compare results to grit-blast/sol-gel, results of the nylon pad process tested at 140°F and 95-100% RH were required.

Al 2024-T3 wedge test adherends were cleaned with acetone-soaked, lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company medium Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Abraded panels were coated with Boegel-EPII solution within 10 minutes of deoxidation in all cases. Adherends were dried at ambient laboratory temperature for 30 minutes and primed with BR 6747-1. Primed panels were dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Panels were machined into specimens, measured for bondline thickness, and tested at 120°F and 95-100% RH as well as 140°F and 95-100% RH. Results are shown in Table 32.

Table 32: Effect of Testing Conditions on Nylon Pad Wedge Test Results

Wedge Test Conditions	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
120°F & 95-100% RH	1.10	0.07	0.10	0.11	0.18	0.18	0.21	0.23	97% co
140°F & 95-100% RH	1.09	0.05	0.11	0.15	0.22	0.26	0.29	0.33	86% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.2.14 Initial Bond Strength Results

In order to determine the initial strengths of bonded joints without the effect of moisture conditioning using Boegel-EPII over a nylon pad-abraded surface, tensile lap shear and floating roller peel tests were conducted. This was to ensure the initial strength of the nylon pad-abraded bonded joints treated with Boegel-EPII is similar to that of PAA-prepared bonded joints and analogous to the grit-blast/sol-gel evaluation in section 3.1.7. Adherends composed of Al 2024-T3 were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. The adherends were abraded with 3-inch diameter 3M Company coarse Roloc™ pads using a 20,000-RPM nitrogen-driven grinder. Adherends were blown clean with 35-psi nitrogen prior to application of Boegel-EPII to remove any residual debris from the bond surface. Boegel-EPII solution was applied using an acid brush, keeping the surfaces wet for 3 minutes. Abraded panels were coated with Boegel-EPII solution within 1 minute of deoxidation in all cases. Adherends were dried at ambient laboratory temperature (70°F) for 30 minutes and primed with BR 6747-1. Primed panels were dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Lap shear testing was performed at –65°F, 70°F, and 180°F after a four-minute soak at temperature. Floating roller peel testing was performed at 70°F and –65°F. Published data from 3M Company[18] on AF 163-2M using CAA primed with EC-3917 were used as a control along with PAA panels primed with BR 6747-1 and cured according to the manufacturer's recommendations to compare initial strength results. Results are shown in Table 33. Specimens failed cohesively under each testing condition except –65°F floating roller peel. Those specimens exhibited roughly 80% cohesive failure. Location of the other failure (~20%) appeared to be within the primer as verified through EDS.

Table 33: Initial Bond Strength Results for Nylon Pad-Abraded Specimens Treated with Boegel-EPII and Primed with BR 6747-1

Surface Preparation	Lap Shear Strength (psi) [% Co Failure]			Peel Strength (pli) [% Co Failure]	
	-65°F	70°F	180°F	-65°F	70°F
Nylon-Pad / Boegel EPII / BR 6747-1	5429 [94% Co]	5471 [98% Co]	3934 [97% Co]	58.3 [80% Co]	68.8 [100% Co]
Published data on CAA / EC-3917	6400	5700	3600	58.0	79.0

co: cohesive

Conducting on-aircraft bonded repairs typically requires curing at lower temperatures than recommended by the adhesive manufacturers due to wide temperature spreads caused by "heat sinks" in the structure. Curing under vacuum pressure is also commonplace in the field environment as previously discussed in section 3.2.11. Therefore, several sets of mechanical strength tests were conducted curing 0.06 psf AF 163-2M under 15 inches Hg vacuum pressure in order to replicate field-level bonding conditions. Two adhesive cure cycles were evaluated using vacuum cure pressures, (1) the manufacturer's recommended cycle of 60 minutes at 250°F, and (2) 6 hours at 200°F. The same nylon pad/sol-gel surface preparation used for the positive pressure testing was used for the vacuum testing except the primer cure cycle was varied between precure and cocure (section 3.2.9). Two lap shear and peel panels (10 specimens) were fabricated and tested for each condition, unless otherwise noted. Specimens fabricated using grit-blast/silane and primed with BR 127 were used as a field-level control process. Results of the lap shear testing for specimens cured at 250°F are shown in Table 34. Results of the lap shear testing for specimens cured at 200°F are shown in Table 35. There is no difference in lap shear strength due to surface preparation when using either of the two adhesive cure cycles. However, when the adhesive was cured at 200°F and tested at –65°F, the nylon-pad/sol-gel specimens exhibited large amounts of adhesive failure at the primer-adhesive interface, even when using the primer cocure method.

Table 34: Lap Shear Strength when Cured for 60 Minutes at 250°F and 15 in Hg

Surface Preparation	Bond Primer	Lap Shear Strength (psi) (%cohesive failure)					
		-65°F		70°F		180°F	
Grit-Blast/Silane	BR 127 (precured)	5701	(93% co)	4777	(98% co)	2857	(100% co)
		4617	(93% co)	4179	(98% co)		
Nylon Pad/Sol-Gel	BR 6747-1 (precured)	5102	(86% co)	4196	(100% co)	1808	(97% co)
		5491	(90% co)	4188	(98% co)	3931	(99% co)
Nylon Pad/Sol-Gel	BR 6747-1 (cocured)	5327	(96% co)	5354	(100% co)	3114	(98% co)
		5290	(95% co)	4651	(99% co)		

Table 35: Lap Shear Strength when Cured for 6 Hours at 200°F and 15 in Hg

Surface Preparation	Bond Primer	Lap Shear Strength (psi) (% cohesive failure)		
		-65°F	70°F	180°F
Grit-Blast/Silane	BR 127 (precured)	5473 (93% co)	5793 (100% co)	3548 (99% co)
		4883 (94% co)	5044 (100% co)	
Nylon-Pad/Sol-Gel	BR 6747-1 (precured)	5105 (36% co)	4911 (100% co)	3025 (100% co)
		5504 (93% co)	4827 (100% co)	3374 (100% co)
Nylon-Pad/Sol-Gel	BR 6747-1 (cocured)	4978 (35% co)	5005 (100% co)	3454 (100% co)
		4881 (20% co)	5092 (100% co)	

Results of the floating roller peel testing for panels cured at 250°F for 60 minutes are shown in Table 36. Results of the floating roller peel testing for panels cured at 200°F for 6 hours are shown in Table 37. When tested at ambient temperature (70°F), there appears to be little difference in peel strength due to surface preparation, and all specimens failed cohesively. However, when tested at –65°F, the specimens fail in the primer layer and exhibit lower bond strengths. Although there is a wide range of peel strengths, there does not appear to be any trends associated with surface preparation. Specimens cured at 200°F exhibit lower peel strengths at both test temperatures when compared to specimens cured at 250°F.

Table 36: Floating Roller Peel Strength when Cured for 60 Minutes at 250°F and 15 in Hg

Surface Preparation	Bond Primer	Peel Strength (pli) (% cohesive failure)	
		-65°F	70°F
Grit-Blast/Silane	BR 127 (precured)	64.7 (76% co)	67.9 (98% co)
		47.4 (18% co)	63.5 (100% co)
Nylon-Pad/Sol-Gel	BR 6747-1 (precured)	42.9 (14% co)	64.6 (98% co)
		50.5 (10% co)	68.2 (96% co)
Nylon-Pad/Sol-Gel	BR 6747-1 (cocured)	44.6 (10% co)	68.3 (98% co)
		39.3 (15 % co)	65.2 (97% co)

Table 37: Floating Roller Peel Strength when Cured for 6 Hours at 250°F and 15 in Hg

Surface Preparation	Bond Primer	Peel Strength (pli) (% cohesive failure)	
		-65°F	70°F
Grit-Blast/Silane	BR 127 (precured)	54.6 (90% co)	59.8 (96% co)
		49.7 (60% co)	57.4 (100% co)
Nylon-Pad/Sol-Gel	BR 6747-1 (precured)	36.5 (10% co)	54.9 (97% co)
		37.1 (5% co)	62.4 (100% co)
Nylon-Pad/Sol-Gel	BR 6747-1 (cocured)	32.1 n/r	57.0 (98% co)
		25.8 (10% co)	53.2 (97% co)

3.3 Optimization of Sol-Gel Surface Preparation Using Sandpaper Surface Activation Techniques

The use of grit-blasting and nylon-pad abrading prior to application of Boegel-EPII solution provided adequate bond surfaces for adhesive bonding. Good initial strength and bond durability results were achieved using both deoxidation techniques (sections 3.1 and 3.2), with grit-blasting providing better wedge test results at 140°F and 95-100% RH. Nylon pad deoxidation provides a significant benefit when compared to grit-blasting due to the lack of grit containment required during deoxidation. Sandpaper surface activation techniques were evaluated as another alternative to grit-blasting.

In order to optimize the parameters associated with activating the bonding surface and subsequent application of Boegel-EPII and bond primers, two designed experiments were conducted to determine significant processing factors. Since a major evaluation of processing parameters was conducted on the nylon pad deoxidation process, a similar evaluation was not conducted using sandpaper deoxidation. Upon completion of the two designed experiments, initial strength testing was performed on bonded joints prepared with the resulting "optimal" process.

3.3.1 Sandpaper Deoxidation Designed Experiment #1

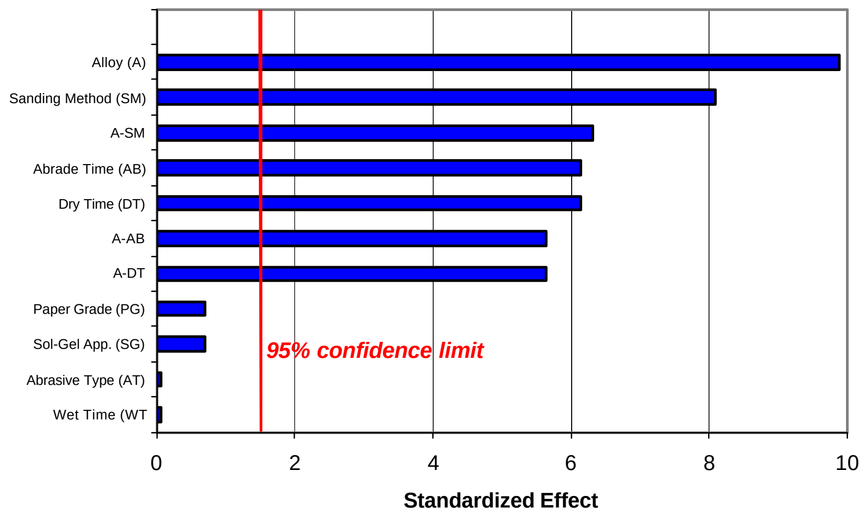
The first of two designed experiments evaluated eight processing factors as shown in Table 38. An L16 test matrix was designed and conducted to evaluate processing factors and interactions associated with performing a surface preparation using sandpaper to deoxidize the surface prior to application of Boegel-EPII solution.

Both Al 2024-T3 and Al 7075-T6 aluminum alloys were evaluated since both 2000 and 7000 series alloys are used in many aircraft applications and may differ significantly in their responses to sandpaper abrasion due to different hardness and alloying elements. All adherends were cleaned with acetone and wiped with lint-free wipes until no remaining trace of grease, dirt, or contamination was visibly present. Two types of abrasive paper were used in this experiment, Craftsman aluminum oxide (Al_2O_3) and Craftsman silicon carbide (SiC), both purchased from Sears. Two grades of abrasive paper were used, fine (220 grit) and coarse (120 grit). Two sanding methods were also used to abrade the panels, a manual or hand sanding method using a sanding block and an air-driven jitterbug (manufactured by National Detroit, Inc.). Panels were abraded for either 2 or 5 minutes. Boegel-EPII solution was brush applied for either 10 or 20 minutes and dried at ambient temperature (70°F) for either 30 or 60 minutes prior to application of primer. Boegel-EPII solution was applied within 10 minutes of deoxidation in all cases. BR 6747-1 primer was applied using a spray gun. Panels were dried at ambient temperature for 30 minutes and cocured with 0.06 psf AF 163-2M adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Wedge test panels were machined into specimens, measured for bondline thickness, and tested at 120°F and 95-100% RH. Results of the designed experiment analysis are shown in Figure 12.

Table 38: Processing Factors for Sandpaper Deoxidation Designed Experiment #1

Factor	Parameter #1	Parameter #2
Alloy	Al 2024-T3	Al 7075-T6
Abrasive type	alumina	SiC
Paper grade	fine	coarse
Sanding method	hand	jitterbug
Abrade time	2 min	5 min
Application method	brush	spray
Boegel EPII wet time	10 minutes	20 minutes
Dry time	30 minutes	60 minutes

Figure 12: Significance of Sandpaper Deoxidation Processing Factors for the Sandpaper Designed Experiment #1



Four processing factors were deemed to be significant to the 95% confidence limit using failure mode as the evaluating criterion: alloy type, sanding method, abrade time, and Boegel-EPII dry time (Table 39). Experiments previously conducted using nylon-pad surface activation indicated the abrade-time factor (3.2.4) and Boegel-EPII dry-time factor (3.2.7) were insignificant. Although those two factors were determined to be significant in this designed experiment using sandpaper abrasion, it was doubtful that the mere use of sandpaper versus nylon pads caused the two factors to become significant since the two abrasion processes are so similar. Instead, it is more likely that the addition of the four insignificant factors (paper grade, application method, wet time, and abrasive type) resulted in the Anova analysis to be too sensitive to differences in test results due to increasing the number of degrees of freedom in the experiment. The first sandpaper designed experiment also showed that hand sanding provided better results than using the air-driven jitterbug. This did not match expected results since the jitterbug abraded the surface more aggressively and evenly than hand sanding. In any case, the better results gained with hand sanding did provide yet another option to field-level maintainers to perform an on-aircraft bonded repair without the use of specialized equipment. However, hand sanding would be a difficult process to control due to the lack of quantifiable factors such as pressure and speed resulting in greater variability from application to application.

Table 39: Optimum Processing Parameters for Sandpaper Designed Experiment #1

Factor	Optimum Parameter
Alloy	Al 2024-T3
Sanding method	hand
Abrade time	2 minutes
Dry time	4 hours
Paper grade	Insignificant
Application method	Insignificant
Boegel EPII wet time	Insignificant
Abrasive type	Insignificant

Alloy type was the most significant factor. Al 2024-T3 wedge test panels performed better than Al 7075-T6 wedge test. This was likely due to the difference in hardness between the two alloys. Al 2024-T3 has a Brinell hardness[19] of 120 Bhn[20] compared to the Brinell hardness of Al 7075-T6 of 150 Bhn[21]. The softer of the two alloys, Al 2024-T3, would be easier to abrade, thus yielding a rougher surface. The rest of the processing factors were deemed to be insignificant. The results of wedge test specimens processed with the “optimum” processing parameters from

Table 39 are shown in Table 40 and compared to the results of the worst-performing wedge test specimens. Even with “optimum” processing conditions, the experiment was unable to provide failure modes above 95% cohesive. Due to the inferior results of the “optimized process” from the first designed experiment compared to nylon pad/sol-gel, a second designed experiment was conducted using different processing parameters in the hopes of finding a better process that yielded improved results.

Table 40: Comparison of Wedge Test Results from Sandpaper Designed Experiment #1

Sandpaper Process	Initial (in)	Cummulative Crack Growth (in)					Failure Mode*
		1 hr	8 hr	24 hr	7 days	28 days	
Optimum	1.12	0.03	0.05	0.12	0.16	0.21	94% co
Worst Performing	1.13	0.05	0.06	0.12	0.26	0.74	0% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.3.2 Sandpaper Deoxidation Designed Experiment #2

The second sandpaper designed experiment evaluated several different processing factors (Table 41). Instead of evaluating different sanding methods as in the first designed experiment, a pneumatic 5-inch diameter random orbital sander (Dynabrade Model 57016) was used to abrade the surface for all wedge test panels. A single type of abrasive paper was used for this evaluation, Norton Company™ 5-inch diameter self-sticking sanding discs. Two different solvents were used to degrease adherends, acetone and isopropyl alcohol (IPA). Two grit sizes of aluminum oxide were used for abrading, 220-grit and 120-grit. Another processing factor evaluated whether solvent wiping the bond surface after the sanding step provided any benefit. The amount of time between the sanding step and application of Boegel-EPII solution (post-deoxidation time) was varied for an experimental factor to determine if a sanded surface had a maximum activated life. Two separate Cytec bond primers were used in this experiment, waterborne, chromated BR 6747-1, and waterborne, nonchromated BR 6757-1. Primer was applied using two different methods, spray using an HVLP gun and manual wiping using a lint-free cloth. Finally, panels were cured using either positive pressure or vacuum pressure. These processing factors and their interactions were all evaluated using an L16 test matrix that was composed of sixteen wedge test panels. All wedge test specimens were conditioned at 120°F and 95-100% RH.

Wedge test adherends were cleaned with the specified solvents in order to remove any contamination from the surface and then abraded with 3-inch 3M Company Scotch-Brite™ Roloc™ fine pads to obtain a baseline surface to begin the process. Adherends were solvent wiped again and deoxidized with Al_2O_3 sandpaper using the random orbital sander. Clean, dry nitrogen was used to operate the pneumatic orbital sander at a pressure of 50 psi. Compressed nitrogen was used to remove any debris remaining on the surface after deoxidation. Boegel-EPII solution was applied using an acid brush, and the surface was kept wet for 3 minutes. The adherends were dried at ambient laboratory conditions (70°F and 60% RH) for 30 minutes. After 30 minutes had elapsed, primer was applied to a nominal thickness of 0.1-0.3 mil, and dried at ambient temperature for 30 minutes. The primer was cocured with 0.06 psf AF 163-2M adhesive in a portable autoclave for 60 minutes at 250°F and either 20 inches Hg vacuum pressure or 35 psi positive pressure. Cured wedge test panels were machined into specimens and measured for bondline thickness using an optical microscope. All specimens were tested at 120°F and 95-100% RH for 28 days.

Table 41: Processing Factors and Levels for Sandpaper Designed Experiment #2

Factor	**Level #1**	**Level #2**
Solvent type	Isopropyl Alcohol	Acetone
Sandpaper grit	220	120
Solvent wipe after deoxidization	no	yes
Post-deoxidization time	<10 min	60 min
Primer type	BR 6757-1	BR 6747-1
Primer application method	cloth	HVLP spray gun
Cure pressure	vacuum	positive pressure

Crack length measurements were used to determine the significance of factors and interactions to the 95% confidence limit using a design of experiments philosophy. Factors that exhibited a standardized effect greater than the 95% confidence limit were considered significant as shown in Figure 13. Three factors and two interactions were deemed to be significant. The three significant factors were primer type, primer application method, and cure pressure. Only one of the significant interactions could be identified, the interaction between the primer application method and the cure pressure. The other significant interaction term was unidentifiable because the designed experiment compounded two interaction terms. Two possible interactions existed and none of the factors contained in those interactions were deemed to be significant in the experiment by themselves. No method of determining which of the possible interactions was actually significant existed. The “optimum” set of processing parameters for this experiment included spray-applied BR 6747-1 primer and vacuum-cure. The specimens that were processed using a bond primer (spray applied) and cured under vacuum exhibited the highest percentage of cohesive failure and the smallest amount of crack growth. Results of the best and worst performing wedge test panels from this experiment are shown in Table 42. There is a drastic difference in wedge test performance due to the significant processing factors and interactions evaluated in this designed experiment. All other processing factors and interactions were considered to be insignificant. These included solvent type, sandpaper grit, solvent wipe after sanding, and time between sanding and application of Boegel-EPII solution.

Figure 13: Significance of Processing Factors and Interactions for the Second Sandpaper Designed Experiment

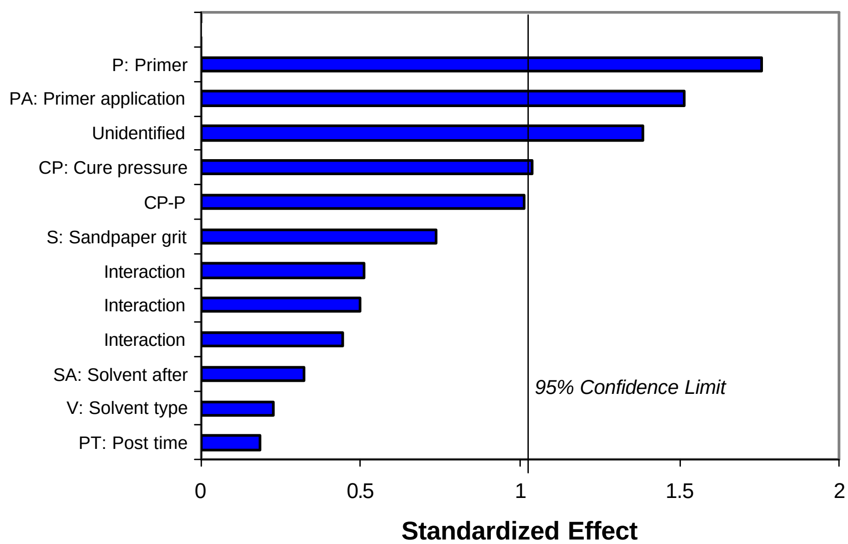


Table 42: Comparison of Wedge Test Panels from Sandpaper Designed Experiment #2

Sandpaper Process	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Optimum	1.15	0.05	0.09	0.13	0.14	0.14	0.15	0.15	96% co
Worst Performing	1.20	0.07	0.09	0.15	0.23	0.45	0.57	0.69	0% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Wedge test specimens primed with BR 6757-1 exhibited interfacial failure modes between the primer and metal. Wedge test specimens primed with BR 6747-1 exhibited a higher percentage of cohesive failure and shorter crack lengths than the specimens primed with BR 6757-1. A number of the specimens primed with BR 6747-1 exhibited 90-95% cohesive failure versus wedge test specimens exhibiting complete adhesive failure when primed with BR 6757-1.

Priming using the cloth-wipe process proved to be detrimental to wedge test performance. When the primer was applied with the cloth, specimens exhibited interfacial failure modes as shown in Figure 14. The experiment showed that specimens primed using a HVLP spray gun exhibited higher amounts of cohesive failure. This proved to be especially true for panels that were primed with BR 6747-1 since the specimens primed with BR 6757-1 exhibited interfacial failure modes regardless of the primer application method. It is possible that the 30-minute ambient temperature dry time was insufficient to fully dry the Boegel-EPII coating prior to application of the primer. The sol-gel coating may have been damaged or partially removed while wiping the waterborne primer. This same problem might not be noticed with a spray application of the primer since this deposits the primer on the surface without touching the bond surface.

Figure 14: Failure Mode Exhibited Using Cloth-Applied Primer

Wedge test specimens cured under vacuum exhibited better results than specimens cured with a positive pressure cure cycle. The best-performing wedge test specimens were cured under vacuum and primed with BR 6747-1 using an HVLP gun. When comparing those vacuum-cured specimens to similar specimens primed with BR 6747-1 using an HVLP spray gun and cured under positive pressure, the vacuum-cured specimens' failure modes were slightly more cohesive. The optimum vacuum-cured specimens exhibited 95% cohesive failures. The remaining 5% interfacial failure (between the primer and metal) occurred at the edges of the specimen. The positive-pressure wedge specimens exhibited approximately 90% cohesive failure with the small amount of interfacial failure occurring at the edges. However, the nicks at the edges of the specimens cured under positive pressure were larger than the nicks of the specimens cured under vacuum. Curing the adhesive under vacuum caused the formation of porosity within the adhesive bondline. Trapped porosity weakens the mechanical properties of the adhesive and reduces the amount of stress at the interface. When the wedge test specimens are cured with positive pressure, the adhesive exhibits stronger mechanical properties due to the lack of porosity. Since the adhesive is stronger, the crack tends to stress the interface more, which can lead to more interfacial failure in the wedge test specimens. It was seen in this experiment that the crack growth in the vacuum-cured specimens was slightly higher than the specimens cured with positive pressure. Although the vacuum-pressure cure cycles appear to provide a benefit in the wedge test for the sol-gel surface preparation, it is not a valid comparison to the positive-pressure-cured specimens since the properties of the adhesive are not the same in both tests. The purpose of evaluating the cure pressure was to ensure that curing under vacuum pressure did not lead to a detrimental effect on the durability of the bond. Vacuum cure cycles are typically used in the field for on-aircraft repairs due to the difficulty associated with applying positive pressure on aircraft.

3.3.3 Initial Strength Test Results

Specimens were fabricated with Al 2024-T3 adherends for tensile lap shear and floating roller peel testing at ambient temperature. Specimens deoxidized with sandpaper, treated with Boegel-EPII solution, and primed with BR 6747-1 using the “optimized” process derived from the second designed experiment were compared to specimens prepared with PAA and primed with BR 127.

Adherends were cleaned with acetone in order to remove organic contamination from the surface. The wedge test panels were abraded with 3-inch 3M Company Scotch-Brite™ Roloc™ fine pads to obtain a baseline surface to begin the surface preparation. Wedge test panels were solvent wiped again and deoxidized with varying grits of Al_2O_3 sandpaper using a random orbital sander. Clean, dry nitrogen was used to operate the pneumatic orbital sander at a pressure of 50 psi in order to prevent surface contamination from dirty and oily compressed air lines. Compressed nitrogen was used to remove debris remaining on the surface after deoxidation. Boegel-EPII solution was applied using an acid brush, and the surface was kept wet for 3 minutes. The wedge test panels were dried at ambient laboratory conditions (70°F and 60% RH) for 30 minutes. After drying, primer was applied to a nominal thickness of 0.1-0.3 mils and dried at ambient temperature for 30 minutes. The primer was cocured with 0.06 psf AF 163-2M adhesive in a portable autoclave for 60 minutes at 250°F and either 20 inches Hg vacuum pressure or 35 psi positive pressure. Cured panels were machined into specimens and measured for bondline thickness prior to testing using an optical microscope. Results for are shown in Table 43.

Table 43: Ambient Temperature Initial Bond Strength Results for Sandpaper-Abraded Specimens Treated with Boegel-EPII and Primed with BR 6747-1

Surface Prep	**Lap Shear (PSI)**		**Peel (PLI)**	
	Vacuum	***Positive Pressure***	***Vacuum***	***Positive Pressure***
PAA / BR 127	3060	5816	60.9	53.4
Sandpaper / Boegel-EPII	3191	6077	63.3	60.2

All failure modes were cohesive and all the strengths were very similar for specimens prepared with PAA/BR 127 and sandpaper deoxidation/Boegel-EPII/BR 6747-1 treatment. The large reduction in lap shear strength when cured under vacuum pressure also explains the improved wedge test results due to vacuum curing discussed in Section 3.3.2.

3.4 Evaluation of Laser Deoxidation Process

Craig Walters Associates (CWA), Dublin, OH, developed prototype lasers for an environmentally friendly alternative for paint stripping in aircraft applications[22]. They also wanted to demonstrate the feasibility of using lasers to deoxidize and texturize surfaces for adhesive bonding applications. The laser utilized by Craig Walters Associates was a Nd:YAG Big Sky Laser Technologies Model CFR 200-20. The wavelength of the pulses was 1064-nm. Pulses were delivered through the fiber at 20 Hz with pulse widths in the 15- to 25-ns range and energy per pulse up to 200 mJ. Two experiments were conducted over two separate trips to CWA. Boegel-EPII solution was used following laser deoxidation for preparation of the bonding surfaces. Wedge crack extension testing was performed to screen the process variables. Since the motorized table that supported the wedge test adherends was of limited size, 3.5-inch by 6-inch adherends were used for this experiment, yielding three 1-inch wide wedge test specimens per panel.

3.4.1 Craig Walters Associates Evaluation #1

CWA determined the laser settings for the first experiment. During the first experiment, a nearly flat-top beam spatial profile was used. Two laser fluence levels were evaluated, a level intended for texturizing (1.4 J/cm^2) and a level intended for texturizing and deoxidizing (2.5 J/cm^2). Control data were generated via grit-blasting with 50-micron aluminum-oxide. Two surface preparations were evaluated after deoxidation, silane treatment with BR 127 primer as used in the GBS preparation and Boegel-EPII sol-gel solution with BR 6747-1 primer. The purpose of using a silane treatment was to determine if using the laser to texturize a grit-blasted surface would improve the results in the wedge test when tested at 140°F and 95-100% RH. In past experiments, GBS performs well in wedge tests when conditioned at 120°F and 95-100% RH but fail when conditioned at 140°F and 94-100% RH.

Al 2024-T3 adherends were degreased with acetone-soaked, lint-free cloths prior to deoxidation. Those to be prepared using silane were grit-blasted. All wedge test adherends were laser treated using one of the two fluence levels. Silane application for 10 minutes or Boegel-EPII solution application for 3 minutes followed. Adherends were blown dry with 35-psi nitrogen. Silane-treated panels were dried at 200°F for 60 minutes prior to priming with BR 127. Boegel-EPII-treated adherends were dried for 30 minutes at ambient temperature (70°F) and primed with BR 6747-1. Both primers were spray applied and cured for 60 minutes at 250°F in an air-circulating oven. Treated adherends were bonded with 0.06 psf AF 163-2M and cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Specimens were tested at either 120°F and 95-100% RH or 140°F and 95-100% RH. Control wedge test panels were fabricated using the optimized GBS surface preparation and grit-blast/Boegel-EPII process. GBS wedge test specimens were tested at 120°F and 95-100% RH in order to ensure the silane surface preparations were conducted correctly. Sol-gel wedge test specimens were tested at 140°F and 95-100% RH. Results of the control wedge data are shown in Table 44. It should be noted that the GBS results (90% cohesive failure) are normally expected to fail 100% cohesively after 28 days at 120°F and 95-100% RH.

Table 44: Control Wedge Test Data

Process	Test Condition	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
			1 hr	8 hr	24 hr	7 days	21 days	28 days	
Grit-blast / silane / BR 127	120°F & 95-100% RH	1.16	0.05	0.07	0.07	0.11	0.12	0.13	90% Co
Grit-blast / Boegel EPII / BR 6747-1	140°F & 95-100% RH	1.13	0.03	0.07	0.10	0.17	0.22	0.24	95% Co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Results using the laser to texturize the surface (1.4 J/cm^2) are shown in Table 45. The specimens prepared with both the silane and Boegel-EPII processes exhibited adhesive failure modes. Results using the laser to texturize and deoxidize (2.5 J/ cm^2) are shown in Table 46. Although the higher power setting used with Boegel-EPII improved performance in the wedge test, specimens still exhibited mostly adhesive failure. Further investigation into the results determined that the bondlines of the laser-exposed specimens were much thinner than those of the control specimens. Table 47 displays the correlation between bondline thickness and failure mode. Specimens with bondlines thinner than 0.003-inch (3.0 mils) exhibited higher amount of adhesive failure. The targeted bondline thickness was 0.005-inch (5.0 mils). Since bondlines were thin, another round of testing was required to generate data with acceptable bondline thicknesses.

Table 45: Effect of Using the Laser to Texturize (1.4 J/cm²) the Bonding Surface

Process	Test Condition	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
			1 hr	8 hr	24 hr	7 days	21 days	28 days	
Grit-blast/Laser/Silane/BR 127	140°F & 95-100% RH	1.30	0.06	0.14	0.17	0.25	0.32	0.32	20% Co
Laser / Boegel EPII / BR 6747-1	140°F & 95-100% RH	1.24	0.13	0.19	0.25	0.36	0.47	0.51	0% Co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Table 46: Effect of Using the Laser to Texturize and Deoxidize (2.5 J/cm²) Bonding Surface

Process	Test Condition	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
			1 hr	8 hr	24 hr	7 days	21 days	28 days	
Laser / Silane / BR 127	120°F & 95-100% RH	1.20	0.13	0.15	0.22	0.27	0.32	0.36	13% Co
Laser / Boegel EPII / BR 6747-1	140°F & 95-100% RH	1.24	0.09	0.14	0.15	0.24	0.30	0.30	43% Co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Table 47: Comparison Between Bondline Thickness and Failure Mode

Deoxidation Process	Fluence (J/cm²)	Surface Treatment	Bondline Thickness (mils)	Failure Mode
Grit-blast	n/a	Silane	4.6	90% co
Grit-blast	n/a	Boegel EPII	4.6	95% co
Grit-blast / Laser	1.4	Silane	2.4	20% co
Laser	1.4	Boegel EPII	2.4	0% co
Laser	2.5	Silane	2.1	13% co
Laser	2.5	Boegel EPII	2.9	43% co

co: cohesive failure

3.4.2 Craig Walters Associates Evaluation #2

During the second laser experiment, two types of beam profiles were evaluated: the flat-top profile used in the first experiment and a near-Gaussian profile. A concerted effort was made to control bondline thickness to the target value of 5 mils. In an effort to improve the wedge test performance or further evaluate processing factors, the following changes were made to the Boegel-EPII process:

1. upon completion of the 3-minute Boegel-EPII application, the adherends were dried at ambient conditions (70°F and 60% RH) for 30 minutes, and
2. the BR 6747-1 primer was cocured with the adhesive.

In addition, one set of specimens was fabricated using BR 6757-1 nonchromated bond primer. Several variables were evaluated in this second experiment including laser profile, alloy (Al 2024-T3 versus Al 7075-T6), alloy cladding (clad versus bare), and bond primer (BR 6747-1 and BR 6757-1). Bondline thickness was successfully controlled to 5 mils ± 1 mil. All test panels were bonded with 0.06 psf AF 163-2M and cured for 60 minutes at 250°F and 35-40 psi. Specimens were tested at 120°F and 95-100% RH. All results, shown in Table 48, appear to be much improved from the first experiment. This is likely due to better control over bondline thickness and cocuring of the bond primers with the adhesive.

Table 48: Wedge Test Results for Laser Deoxidation Evaluation #2

Materials	Laser Profile	Initial (in)	Cummulative Crack Growth (in)						Failure Mode
			1 hr	8 hr	24 hr	7 days	21 days	28 days	
Al 2024-T3 / BR 6747-1	n/a-grit-blast	1.09	0.01	0.09	0.09	0.13	0.16	0.17	98% co
Al 2024-T3 / BR 6747-1	Flat top	1.19	0.03	0.06	0.06	0.13	0.15	0.15	98% co
Al 2024-T3 / BR 6747-1	Gaussian	1.18	0.01	0.08	0.09	0.12	0.16	0.19	96% co
Al 2024-T3-clad / BR 6747-1	Flat top	1.09	0.01	0.12	0.14	0.16	0.18	0.18	95% co
Al 2024-T3-clad / BR 6747-1	Gaussian	1.15	0.03	0.09	0.12	0.14	0.15	0.17	97% co
Al 7075-T6 / BR 6747-1	Gaussian	1.21	0.02	0.09	0.11	0.11	0.13	0.13	80% co*
Al 2024-T3 / BR 6757-1	Gaussian	1.18	0.00	0.07	0.08	0.08	0.11	0.11	97% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.5 Evaluation of Boegel-EPII Sol-Gel Solution Parameters

Although the Boeing Company provided details on mixing Boegel-EPII and recommended suppliers for the chemicals, AFRL requested that UDRI conduct several experiments to determine the significance of parameters such as GTMS manufacturer, simplified mixing procedures, constant mixing versus occasional shaking, and the maximum pot life of Boegel-EPII. This section documents those experiments.

3.5.1 Effect of GTMS Manufacturer

Gelest, Inc. and Dow Corning, Inc. are two of several companies that manufacture γ-glycidoxypropyltrimethoxysilane (GTMS). Batches of Boegel-EPII solution were prepared using standard mixing procedures (Table 1) with Dow Corning Z-6040 and Gelest SIG5840.0. Wedge test panels were fabricated using both grit-blast and nylon-pad surface activation methods and Boegel-EPII mixed with GTMS from the two manufacturers. A total of four wedge test panels were fabricated with Al 2024-T3, representing one panel for each condition above.

Adherends were solvent wiped with acetone until clean and deoxidized either by abrading with 3M Company Roloc™ 3-inch diameter medium pads or grit-blasting with 50 micron aluminum oxide. Once deoxidized, adherends were blown with clean, dry, compressed nitrogen to remove residual grit or debris. Boegel-EPII was applied using an acid brush within 30 minutes of the deoxidation process. The surfaces were kept wet for 3 minutes, and the panels were orientated vertically to drain and dry at ambient temperature (70°F) for 30 minutes. Cytec BR 6747-1 bond primer was applied with a spray gun according to the manufacturer’s recommendations. Once primed, adherends were dried at ambient temperature for 30 minutes. Primer on the grit-blasted panels was precured for 60 minutes at 250°F. Adhesive was applied to the nylon pad-abraded panels upon completion of the 30-minute ambient-temperature dry so the primer and adhesive would be cocured. AF 163-2M (0.06 psf) adhesive was used to bond both the grit-blasted and nylon pad-abraded panels. The adhesive was cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Specimens were tested at 140°F and 95-100% RH. Results are shown in Table 49. There did not appear to be any difference in wedge test results due to the GTMS source. The crack growth and failure modes of specimens deoxidized in the same manner appeared to be equivalent.

Table 49: Effect of GTMS Manufacturer on Wedge Test Results

Deox Step	GTMS Source	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
			1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Grit-blast	Dow Corning	1.14	0.03	0.07	0.10	0.19	0.25	0.27	0.29	99% co
	Gelest	1.16	0.02	0.03	0.05	0.17	0.22	0.24	0.25	98% co
3M Roloc Medium	Dow Corning	1.12	0.02	0.07	0.10	0.22	0.26	0.28	0.30	88% co
	Gelest	1.07	0.00	0.01	0.06	0.20	0.26	0.26	0.29	86% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.5.2 Effect of Simplified Boegel-EPII Mixing Process

The standard mixing instructions for Boegel-EPII are listed in Table 1. According to these instructions, upon mixing the n-zirconium propanol (TPOZ) and glacial acetic acid (GAA), the mixture should be allowed to sit for 10-15 minutes (step #1). Upon the recommendation of Boeing, a simplified Boegel mixing process was developed in which the TPOZ and GAA mixture only required 1-3 minutes to stabilize prior to stirring with the water/silane mixture. Wedge crack extension tests were performed using both the standard and simplified mixing processes.

Al 2024-T3 adherends were solvent degreased with acetone-soaked, lint-free cloths until clean. Adherends were deoxidized either with 50 μm Al_2O_3 grit-blast media, Norton #220 aluminum oxide sandpaper, or 3M Company Roloc™ medium nylon pads. Sanded panels were abraded with a random orbital sander. Nylon-pad abrasion was accomplished using a 20,000 RPM nitrogen-driven grinder. Once deoxidized, adherends were blown with clean, dry, compressed nitrogen to remove residual grit or debris. Boegel-EPII was applied using an acid brush within 30 minutes of the deoxidation process. The surfaces were kept wet for 3 minutes, and the panels were orientated vertically to drain and dry at ambient temperature for 30 minutes. Cytec BR 6747-1 bond primer was applied with a spray gun, and the adherends were dried at ambient temperature for 30 minutes. Primer was precured for 60 minutes at 250°F on all the precured panels. A single set of nylon-pad abraded panels was treated with sol-gel, primed, and cocured with the adhesive. AF 163-2M (0.06 psf) adhesive was used to bond all wedge test panels. The adhesive was cured for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Specimens were tested at 140°F and 95-100% RH. Results are shown in Table 50. The wedge test panels processed using the simplified mixing procedure performed as well as panels processed with the standard mixing procedure using all deoxidation processes.

Table 50: Standard Boegel-EPII Mixing versus Simplified Mixing

Deoxidation Step	Mixing	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
			1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Grit-blast	Standard	1.12	0.05	0.06	0.11	0.18	0.22	0.25	0.26	96% co
	Simplified	1.09	0.04	0.10	0.14	0.22	0.28	0.29	0.33	94% co
#220 Sandpaper	Standard	1.18	0.06	0.13	0.27	0.50	0.68	0.74	0.78	17% co
	Simplified	1.09	0.05	0.11	0.15	0.24	0.37	0.47	0.57	25% co
Nylon pad (precure)	Standard	1.13	0.06	0.10	0.13	0.19	0.25	0.29	0.33	68% co
	Simplified	1.19	0.04	0.10	0.14	0.22	0.28	0.34	0.35	81% co
Nylon pad (cocure)	Standard	1.09	0.05	0.11	0.15	0.22	0.26	0.29	0.33	86% co
	Simplified	1.10	0.04	0.09	0.13	0.20	0.28	0.28	0.31	89% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.5.3 Effect of Constant Mixing Versus Occasional Mixing

Magnetic mixers such as those used to constantly mix Boegel-EPII solution in this program may not be available in all situations. Therefore an alternative mixing approach was evaluated. Two batches of sol-gel were prepared using standard sol-gel mixing instructions. One used constant mixing after step # 3. A second batch was prepared by allowing the solution to sit for 30 minutes after combining the TPOZ/GAA mixture with the water/silane mixture (step # 3). The solution was agitated intermittently by shaking the container during the 30-minute induction period. Two wedge test panels were fabricated per mix of Boegel-EPII solution, using nylon pad deoxidation. Nylon-pad deoxidation was used because it tends to be less sensitive to process changes than grit-blasting.

Al 2024-T3 adherends were solvent degreased with acetone-soaked, lint-free cloths until clean. Adherends were deoxidized with 3M Company Roloc™ medium nylon pads using a 20,000-RPM nitrogen-driven grinder. Adherends were then blown with clean, dry, compressed nitrogen to remove residual debris. Boegel-EPII was applied using an acid brush within 30 minutes of the deoxidation process. The surfaces were kept wet for 3 minutes, and the panels were orientated vertically to drain and dry at ambient temperature (70°F) for 30 minutes. Cytec BR 6747-1 bond primer was applied with a spray gun, and the adherends were dried at ambient temperature for 30 minutes. Adhesive was applied to the adherends and the primer was cocured with the AF 163-2M (0.06 psf) adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Specimens were tested at 120°F and 95-100% RH. Results are shown in Table 51. There is little difference in the crack growth after 28 days between wedge test panels processed with different mixing methods for Boegel-EPII solution. The failure modes are also consistently around 92-93% cohesive with the adhesive failure remaining at the primer-metal interface.

Table 51: Occasional Shaking versus Constant Mixing of Boegel-EPII Solution

Mixing	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
Constant	1.00	0.06	0.07	0.07	0.11	0.12	0.13	0.17	92% co
	1.03	0.02	0.02	0.05	0.09	0.09	0.11	0.12	93% co
Occasional	1.03	0.06	0.06	0.07	0.12	0.12	0.15	0.16	92% co
	1.06	0.02	0.02	0.02	0.10	0.10	0.13	0.14	93% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

3.5.4 Pot Life Evaluation

Boeing recommended using Boegel-EPII solution only up to 10 hours once mixed. In order to verify this time limit, a batch of Boegel-EPII was mixed according to the standard mixing procedures. Once the TPOZ / GAA mixture was added to the GTMS / water mixture, a timer was set. Wedge test panels deoxidized with nylon pads were treated with sol-gel at various time intervals ranging from only 15 minutes to 72 hours in order to define an optimal operating window. One wedge test panel (five specimens) per condition was fabricated.

Al 2024-T3 adherends were solvent degreased with acetone-soaked, lint-free cloths until clean. Adherends were deoxidized with 3M Company Roloc™ medium nylon pads using a 20,000-RPM nitrogen-driven grinder. Adherends were then blown with clean, dry, compressed nitrogen to remove residual debris. Boegel-EPII was applied using an acid brush within 30 minutes of the deoxidation process. The surfaces were kept wet for 3 minutes, and the panels were orientated vertically to drain and dry at ambient temperature for 30 minutes. Cytec BR 6747-1 bond primer was applied with a spray gun, and the adherends were dried at ambient temperature (70°F) for 30 minutes. Adhesive was applied to the adherends, and the primer was cocured with the AF 163-2M (0.06 psf) adhesive for 60 minutes at 250°F and 35-40 psi in a portable autoclave. Specimens were tested at 120°F and 95-100% RH. Results are shown in Table 52.

Even though the standard mixing instructions require a minimum of 30 minutes for Boegel-EPII to mix prior to application, nylon-pad-abraded wedge test specimens treated after only 15 minutes of mixing exhibited cohesive failure modes. After 24 hours of mixing, the Boegel-EPII solution was very cloudy, although wedge test results after 24 hours of mixing still exhibited cohesive failures. After 48 hours of mixing the Boegel-EPII solution was white and opaque. However, wedge test results did not appear to decrease substantially and still yielded 92% cohesive failure. After 72 hours of continuous mixing, the Boegel-EPII solution was opaque and contained small particulates. Wedge test results for panels coated with Boegel-EPII after 72 hours of continuous mixing exhibited similar crack growth to that of panels coated with the same Boegel-EPII solution mixed for only 15 minutes. However, the failure modes exhibited by specimens prepared after mixing for 72 hours were only about 85% cohesive. Boegel-EPII solution appeared to provide adequate performance (95% cohesive failure for a nylon pad abraded surface) after 24 hours of continuous mixing. However, this was not recommended due to the cloudier appearance of the solution and the desire to use the appearance of the solution for a quality control measure.

Table 52: Boegel-EPII Pot Life Evaluation Wedge Test Results

Boegel EP II Mixing Time	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
		1 hr	8 hr	24 hr	7 days	14 days	21 days	28 days	
15 minutes	1.13	0.02	0.05	0.07	0.12	0.15	0.17	0.19	97% co
30 minutes	1.16	0.03	0.06	0.09	0.13	0.16	0.16	0.16	96% co
4 hours	1.14	0.05	0.08	0.09	0.14	0.16	0.17	0.18	94% co
8 hours	1.14	0.05	0.09	0.11	0.16	0.19	0.19	0.20	95% co
24 hours	1.21	0.03	0.09	0.10	0.15	0.16	0.18	0.18	95% co
30 hours	1.11	0.04	0.09	0.10	0.16	0.16	0.17	0.19	93% co
48 hours	1.17	0.04	0.09	0.12	0.17	0.19	0.20	0.22	92% co
72 hours	1.17	0.04	0.08	0.10	0.15	0.18	0.18	0.20	85% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

4 EFFECT OF PROCESS VARIABLES USING CHEMAT TECHNOLOGIES, INC. AL 9201 SOL-GEL SOLUTION

Chemat Technologies Inc., Northridge, CA, developed a sol-gel chemistry for treating aluminum alloys and demonstrated its effectiveness for adhesive bonding in a previous contract with the USAF[23]. Chemat currently markets this sol-gel as AL 9201. This material was identified for possible use as an environmentally friendly surface preparation for adhesive bonding of aluminum alloys. Benefits of AL 9201 are that it is a non-hazardous, environmentally friendly one-part system that possesses an ambient-temperature shelf life of six months. The purpose of this test program was to evaluate AL 9201 sol-gel solution as an aluminum surface preparation for field-level bonded repair processes. This included a trip to Chemat at their facility in Northridge, CA, to fabricate wedge test panels using various processes with AL 9201 solution. UDRI personnel conducted a second round of testing at the Air Force Research Laboratory (AFRL) in order to verify results obtained at Chemat.

4.1 Data Generated at Chemat Technologies Inc.

Three surface preparation processes were used to fabricate wedge test panels at Chemat for evaluation of the AL 9201 solution:

1. ***"Overlap" process***: AL 9201 solution was applied then overlapped with Cytec BR 6747-1 primer,
2. ***Mixture process:*** AL 9201 solution was combined with BR 6747-1 primer to form a mixture and applied in a single coat, and
3. ***Grit-blast/silane-type process:*** a process that mimicked the grit-blast/silane process but utilized AL 9201 and BR 6747-1 primer rather than GTMS/water and BR 127 primer.

Chemat personnel recommended the overlap and mixture processes as the optimal approaches for fabricating wedge crack extension test panels with AL 9201 solution. However, since both of these processes included cleaning steps with an alkaline detergent (Brulin 815), UDRI and AFRL engineers suggested a third process, the grit-blast/silane-type process. This approach followed the grit-blast/silane process but used AL 9201 solution instead of a 1% silane/water mixture and was more practical to perform on aircraft. A detailed description of the steps involved in each of the processes is shown in Table 53. Each utilized a grit-blast deoxidation step using 50 micron aluminum oxide grit. Chemat and UDRI personnel fabricated wedge test panels using all three of these processes on Al 2024-T3 adherends.

Table 53: Processes Used to Evaluate Chemat Technologies AL 9201 Solution

Overlap Process	Mixture Process	Grit-blast/Silane-Type Process
◆ Acetone wipe ◆ Grit-blast with 50 micron alumina ◆ Brulin 815 ultrasonic clean for 15 minutes at 140°F ◆ Brush with Brulin 815 to remove grit ◆ Spray rinse with DI water for 5 min ◆ Dry at 212°F for 30 min ◆ Spray drench with Al 9201 for 5 min ◆ Ambient air dry for 30 minutes ◆ Prime with BR 6747-1 ◆ Ambient dry for 30 minutes ◆ Cure for 60 min @ 250°F ◆ Bond with FM 73M for 60 min @ 270°F & 35-40 psi	◆ Acetone wipe ◆ Grit-blast with 50-micron alumina ◆ Brulin 815 ultrasonic clean for 15 minutes at 140°F ◆ Brush with Brulin 815 to remove grit ◆ Spray rinse with DI water for 5 min ◆ Dry at 212°F for 30 min ◆ Prepare hybrid: 70% by wt BR 6747-1, 30% by wt Al 9201 ◆ Allow hybrid to mix for 10 minutes ◆ Spray apply hybrid to a thickness of 0.1-0.3 mils ◆ Ambient dry for 30 minutes ◆ Cure for 60 minutes at 250°F ◆ Bond with FM 73M for 60 min @ 270°F & 35-40 psi	◆ Acetone wipe ◆ Grit-blast with 50-micron alumina ◆ Blow surface with compressed nitrogen ◆ Spray drench with Al 9201 for 5 min ◆ RT air dry for 30 minutes ◆ Prime with BR 6747-1 ◆ Ambient dry for 30 minutes ◆ Cure for 60 minutes at 250°F ◆ Bond with FM 73M for 60 min @ 270°F & 35-40 psi

Brulin 815: alkaline cleaner

Adherends were prepared and bonded in the same day at Chemat's laboratory in Northridge, CA. All panels were bonded with Cytec FM 73M epoxy film adhesive (0.06 psf) for 60 minutes at 270°F and 35-40 psi in a hydraulic press. Cytec suggests a cure temperature of 250°F but Chemat scientists felt the higher temperature would improve the performance of the AL 9201 coating. All panels were taken back to AFRL and machined into 1-inch wide specimens, and the bondline thickness of each specimen was measured using an optical microscope. Half of the specimens from each panel were sent to Chemat for testing and half were tested at AFRL. All specimens were tested at 120°F and 95-100% RH. Data tested at both facilities were included in the results shown in Table 54. The approach that mimicked the grit-blast/silane process resulted in excessive crack lengths and adhesive failure modes after only seven days at 120°F and 95-100% RH. Wedge test specimens prepared with the "overlap" process also yielded excessive crack lengths and exhibited adhesive failures. However, the mixture process yielded cohesive failure modes and excellent crack growth resistance after 28 days at 120°F and 95-100% RH.

4.2 Data Generated at AFRL

Further evaluation of the AL 9201/BR 6747-1 mixture was conducted at AFRL due to the promising results obtained at Chemat. Two sets of wedge test data were generated, one with specimens tested at 120°F and 95-100% RH and another with specimens tested at 140°F and 95-

100% RH. The set tested at 120°F was fabricated and tested prior to specimen fabrication and testing at 140°F. So not all the same process variables were evaluated.

Table 54: Results of AL 9201 Solution First Round Testing

AL 9201 Process	Fabricator	Initial (in)	Cummulative Crack Growth (in)						Failure Mode
			1 hr	8 hr	24 hr	7 days	21 days	28 days	
Mixture	*Chemat*	1.31	0.00	0.00	0.00	0.00	0.04	0.04	100% co
Mixture	*UDRI*	1.21	0.02	0.03	0.03	0.05	0.10	0.12	100% co
Mixture	*Chemat*	1.32	0.02	0.03	0.03	0.03	0.04	0.04	100% co
Mixture	*UDRI*	1.49	0.00	0.01	0.01	0.01	0.01	0.01	100% co
Overlap	*UDRI*	1.37	0.01	0.01	0.14	0.64	N/A*	N/A*	0% co
Overlap	*Chemat*	1.29	0.03	0.04	0.06	0.21	0.43	0.53	0% co
GBS	*Chemat*	1.37	0.00	0.00	0.15	0.94	N/A*	N/A*	0% co

* Specimens removed from humidity due to excessive crack lengths and adhesive failures.
co: cohesive failure

4.2.1 Wedge Test Results at 120°F and 95-100% RH

The main purpose of this evaluation was to verify results obtained at Chemat and determine any differences due to alternate cleaning steps after grit-blasting (blown nitrogen versus Brulin 815), adhesive type (AF 163-2M and FM 73M), and adhesive cure temperature (250°F versus 270°F). On-aircraft grit removal would be easier to accomplish by blowing with nitrogen or clean air rather than the Brulin 815 rinse utilized at Chemat. Two common adhesives were evaluated, Cytec FM 73M and 3M Company AF 163-2M. Both adhesives were 0.06 psf weight with mat carrier. Chemat cured the wedge test panels fabricated at their facility for 60 minutes at 270°F. However, the manufacturers of both adhesives cite the optimum cure cycle to be 60 minutes at 250°F, and lower temperatures are typically required for on-aircraft repair. Therefore, the effect of cure temperature was evaluated. Finally, two wedge test panels were grit-blasted and primed with BR 6747-1, without AL 9201 sol-gel step.

Al 2024-T3 adherends were solvent degreased with acetone-soaked lint free cloths until clean and blasted with 50 μm Al_2O_3 grit. Blasting debris was cleaned using 30-psi clean, dry nitrogen or by soaking in Brulin 815 for 15 minutes at 140°F using light air agitation. Adherends soaked in Brulin 815 were rinsed with DI water for 5 minutes and dried at 212°F for 30 minutes. A 70% BR 6747-1/30% AL 9201 mixture by weight was stirred with a magnetic mixer for 10 minutes prior to use. Adherends were primed with the mixture to a thickness of 0.0001-0.0003 inch (0.1-0.3 mils). Adherends were cured for 60 minutes at 250°F in an air-circulating oven. Adhesive was applied and cured in a portable autoclave for 60 minutes under 35-40 psi at either 250°F or 270°F. Results for specimens tested at 120°F and 95-100% RH are shown in Table 55.

The “control” mixture process utilizing the Brulin 815 cleaning step and 270°F adhesive cure cycle produced passing wedge test specimens when bonded with both FM 73M and AF 163-2M adhesives. Specimens bonded at 250°F also utilized a blown nitrogen-cleaning step after the grit-blast. These specimens exhibited a mix of cohesive and adhesive failure modes when bonded with both adhesives. Therefore, it was impossible to determine whether the reduction in cohesive failure was due to the cleaning step or adhesive cure cycle. Specimens receiving a grit-blast and then primed with BR 6747-1 (with no AL 9201) exhibited long crack growths and complete adhesive failure after 28 days at 120°F and 95-100% RH. Therefore, adding AL 9201 solution to BR 6747-1 primer significantly improved the performance of the bonded joint in the wedge test significantly. The original results obtained at Chemat were verified by the test results in this experiment.

Table 55: 120°F and 95-100% RH Wedge Test Results Using AL 9201/BR 6747-1 Mixture

Process	Adhesive	Adhesive Cure Temperature	Initial (in)	Cummulative Crack Growth (in)						Failure Mode
				1 hr	8 hr	24 hr	7 days	21 days	28 days	
GB, Brulin 815, & AL 9201 / BR 6747-1	*FM 73*	*270°F*	1.27	0.01	0.01	0.01	0.01	0.02	0.09	100% co
GB, Brulin 815, & AL 9201 / BR 6747-1	*AF 163-2M*	*270°F*	1.18	0.03	0.06	0.11	0.13	0.15	0.18	95% co
GB, N_2 blow, & AL 9201 / BR 6747-1	*FM 73*	*250°F*	1.32	0.03	0.04	0.05	0.07	0.09	0.11	84% co
GB, N_2 blow, & AL 9201 / BR 6747-1	*AF 163-2M*	*250°F*	1.22	0.08	0.10	0.11	0.14	0.14	0.17	79% co
GB, N_2 blow, & BR 6747-1	*FM 73*	*250°F*	1.30	0.05	0.07	0.11	0.44	0.77	0.90	0% co
GB, N_2 blow, & BR 6747-1	*AF 163-2M*	*250°F*	1.20	0.10	0.15	0.30	0.50	0.52	0.54	0% co

co: cohesive failure

4.2.2 Wedge Test Results at 140°F and 95-100% RH

Since positive results were obtained using the mixture of BR 6747-1 and AL 9201 solution, a second evaluation was conducted to better evaluate three processing factors:

1. cleaning step after grit-blast (Brulin 815 bath versus nitrogen blast),
2. adhesive (AF 163-2M versus FM 73M), and
3. adhesive cure temperature.

This evaluation was conducted using Al 2024-T3 adherends with 0.06 psf adhesives.

Al 2024-T3 adherends were solvent degreased with acetone-soaked lint free cloths until clean and blasted with 50 μm aluminum oxide grit. Blasting debris was either cleaned using 30-psi clean, dry nitrogen or by soaking in Brulin 815 for 15 minutes at 140°F using light air agitation. Adherends soaked in Brulin 815 were rinsed with DI water for 5 minutes and dried at 212°F for 30 minutes. A mixture composed of 70% BR 6747-1 and 30% AL 9201 by weight was stirred with a magnetic mixer for 10 minutes prior to use. Adherends were primed with the mixture to a thickness of 0.1-0.3 mils, and cured for 60 minutes at 250°F in an air-circulating oven. Adhesive was applied and cured in a portable autoclave for 60 minutes under 35-40 psi at either 250°F or 270°F. Results for specimens tested at 140°F and 95-100% RH are shown in Table 56.

Results indicate there were two significant processing parameters: the cleaning step after grit-blasting and the adhesive that was used to bond the specimens. The Brulin 815 cleaning step after grit-blasting increased the percentage of cohesive failure in the wedge test. This could be due to the Brulin 815 removing more residual grit from the bonding surface than the nitrogen blast was capable of removing. Secondly, an increase in cohesive failure mode was detected when bonding specimens with FM 73M versus AF 163-2M. This could be due to the increased initial crack length of specimens bonded with FM 73M or due to the fact that AF 163-2M typically exhibits better hot/wet properties than FM 73M[24]. The difference in adhesive cure temperature (270°F versus 250°F) did not seem to make a difference in either crack growth or failure mode for specimens bonded with either adhesive.

Table 56: Wedge Test Results at 140°F and 95-100% RH Using AL 9201/BR 6747-1 Mixture

Clean after GB	Adhesive	Adhesive Cure Temperature	Initial (in)	Cummulative Crack Growth (in)							Failure Mode*
				1 hr	8 hr	24 hr	7 day	14 day	21 day	28 day	
Brulin 815	AF 163-2M	270°F	1.13	0.03	0.08	0.17	0.25	0.25	0.27	0.27	90% Co
Brulin 815	AF 163-2M	250°F	1.13	0.04	0.05	0.13	0.18	0.22	0.22	0.23	94% Co
N_2	AF 163-2M	270°F	1.11	0.03	0.07	0.16	0.22	0.24	0.25	0.28	79% Co
N_2	AF 163-2M	250°F	1.08	0.03	0.11	0.17	0.22	0.26	0.28	0.29	64% Co
Brulin 815	FM 73M	270°F	1.26	0.04	0.04	0.04	0.06	0.07	0.07	0.08	100% Co
Brulin 815	FM 73M	250°F	1.30	0.03	0.04	0.04	0.06	0.09	0.11	0.13	100% Co
N_2	FM 73M	270°F	1.27	0.03	0.05	0.06	0.14	0.16	0.16	0.17	97% Co
N_2	FM 73M	250°F	1.29	0.05	0.05	0.06	0.13	0.16	0.18	0.20	93% Co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

5 DISCUSSION OF RESULTS

5.1 Boegel-EPII Solution Process Results

5.1.1 Grit-blast Surface Activation Process Evaluation

The grit-blast/Boegel-EPII process provided excellent wedge test results. The following bullets summarize the results of the grit-blast process evaluation with Boegel-EPII solution when using wedge test results as the determining criteria.

- Specimens prepared with the grit-blast/Boegel EP II process and bonded with 0.06 psf AF 163-2M passed the wedge test (nearly 100% cohesive failure) when tested at 120°F and 95-100% RH and 140°F and 95-100% RH.
- Specimens primed with Cytec BR 6747-1 waterborne, chromated primer provided the best results.
- Specimens primed with BR 6747-1 passed the wedge test when cured according to the manufacturer's recommendations and when cocured with the adhesive in a single cure cycle at 250°F.
- Cytec BR 127 (MEK-based, chromated) and BR 6757-1 (waterborne, nonchromated) primers did not provide adequate results in the wedge test when used in conjunction with Boegel-EPII solution.
- Specimens failed cohesively in the wedge test when bonded with a number of 250°F-curing, 0.06 psf-weight, mat carrier film adhesives including: 3M Company AF 163-2M, Cytec FM 73M, and Loctite Hysol EA 9628 and EA 9696.
- Boegel-EPII wet times between 2 and 20 minutes were equivalent
- Several sol-gel dry methods yielded acceptable results for initial strength and moisture durability (wedge testing), including ambient drying (between 4 minutes to 4 hours) and force drying with clean, dry nitrogen at pressures between 5-30 psi.
- The grit-blast/Boegel-EPII process worked well on Al 2024-T3 and Al 7075-T6 alloys as well as bare and clad aluminum alloys.

5.1.2 Nylon Pad Surface Activation Process Evaluation

An extensive effort was undertaken to develop a nongrit-blast process for use with the Boegel-EPII sol-gel coating. This was due to the desire of field and depot-level maintenance personnel to find a replacement for grit-blasting due to the grit containment and cleanup issues. The experiments reported in section 3.2 resulted in the following conclusions.

- Specimens prepared with the nylon pad/Boegel EP II process and bonded with 0.06 psf AF 163-2M passed the wedge test (greater than 95% cohesive failure) when tested at 120°F and 95-100% RH but exhibited less amounts of cohesive failure (~85-90% cohesive) when tested at 140°F and 95-100% RH.
- IPA, MEK, and acetone were all compatible solvents for degreasing during the nylon pad/Boegel-EPII process. However, the effectiveness of each of these solvents to remove gross organic contamination from the bond surface was not evaluated since all solvent cleaning was performed on laboratory specimens.
- In a comparison between Standard Abrasives "Buff and Blend" and 3M Company Roloc™ nylon pads, the 3M medium and coarse Roloc™ pads outperformed all other nylon pads.
- No difference was detected for the time required for abrading the bonding surface with a nylon pad between 30 seconds and 2 minutes.
- In a laboratory environment, the time between nylon pad abrasion and application of Boegel-EPII solution was insignificant for times between 1 minute and 2 hours, with all wedge test specimens exhibiting nearly cohesive failure modes.
- No differences were detected in the wedge test due to wetting the bonding surface with Boegel-EPII solution between 2 and 20 minutes.
- Drying the Boegel-EPII solution was accomplished via freestanding ambient drying and force-drying using pressurized, dry clean nitrogen with no change in bond performance. Using air pressures above 40 psi degraded bond performance in the wedge test. It is expected that air pressure versus standoff distance could also be a crucial variable but it was not evaluated in this program.
- Cytec BR 6747-1 primer was applied via two methods, spray and wipe. When primer was applied with a lint-free cloth, bonds exhibited adhesive failure. Specimens

primed using a spray gun exhibited cohesive failures. It must be noted that the wipe-apply method was not optimized.

- Primer was cured using three different methods: precured according to the manufacturer's recommendations, cocured with the adhesive and, fused prior to cocure with the adhesive. The primer fuse and cocure processes exhibited the best results, however cohesive failure modes were detected in at least one experiment when the primer was precured (section 3.2.9).
- No difference was detected when using either Al 2024-T3 or Al 7075-T6.
- No difference was detected when using either positive pressure or vacuum cure cycles.
- Specimens bonded with 3M Company AF 163-2M, Loctite Hysol EA 9628, and EA 9696 all exhibited cohesive failures when using a nylon pad abrasion technique in conjunction with cocured BR 6747-1. Specimens bonded with Cytec FM 73 exhibited cohesive failures intermittently.
- It was observed that water-break tests were more difficult to conduct on nylon pad-abraded surfaces, thus making inspection for contamination more difficult than for grit-blast surfaces.

5.1.3 Results of Sandpaper Surface Activation Process Evaluation

Sandpaper abrasion was evaluated as an alternative to abrading with nylon pads. Two designed experiments were conducted to identify significant processing factors. The following statements summarize the results obtained from those experiments.

- Abrasion using sandpaper provided wedge test results similar to nylon pad deoxidation, but was unable to attain similar results as grit-blast when tested at 140°F and 95-100% RH.
- No difference was detected between using acetone or isopropyl alcohol for solvent degreasing. However, the effectiveness of each of these solvents to remove gross organic contamination from the bond surface was never evaluated since all solvent cleaning was performed on laboratory specimens.

- A solvent wipe after abrasion and prior to application of Boegel-EPII solution had no effect on bond durability when compared to using pressurized nitrogen or air to remove debris.
- The grades (120-220 grit) and types of abrasive paper (alumina and SiC) in this experiment had no effect on bond performance.
- Sanding using an air-driven jitterbug tended to exhibit worse results than hand sanding. Abrading with an air-driven random orbital sander exhibited the best overall results of all the abrading methods evaluated.
- The amount of time between the abrasion and Boegel-EPII application steps was insignificant for times between 1-60 minutes since wedge test specimens failed nearly cohesively.
- Boegel-EPII solution could be applied via brush or spray with no effect on wedge test results.
- Varying the wetting time for Boegel-EPII solution between 10-20 minutes had no effect.
- Specimens primed with Cytec BR 6757-1 performed worse in the wedge test than specimens primed with BR 6747-1.
- Specimens exhibited better results in the wedge test when bond primer was applied with a spray gun versus application with a lint-free cloth.
- Al 2024-T3 specimens exhibited better failure modes than Al 7075-T6 specimens. This was likely due to the increased hardness of Al 7075-T6.

5.1.4 Laser Surface Activation Process Evaluation

Results from the initial laser test matrix completed at Craig Walters Associates were inconclusive due to the thin bondlines witnessed in the wedge test specimens. The thin bondlines caused excessive interfacial failure and no real conclusions could be drawn from any of the data.

The second test matrix evaluated several factors including alloy, bond primer, and laser profile. These specimens exhibited bondline thicknesses on the range of 4-6 mils (0.004-0.006 inch). All specimens exhibited excellent results in the wedge test proving that the following variables were insignificant:

- Flat-top versus Gaussian laser beam profile,
- Al 2024-T3 bare versus Al 7075-T6 bare,
- Al 2024-T3 bare versus Al 2024-T3 clad, and
- BR 6747-1 primer versus BR 6757-1 primer.

The laser provided a textured and clean surface for adhesive bonding that resulted in excellent wedge test results when used in conjunction with Boegel-EPII.

5.1.5 Boegel-EPII Solution Parameter Evaluation

Boegel-EPII solution parameter evaluation results are summarized in the bulleted list below. All statements are based upon wedge test data conducted in section 3.5.

- There was no apparent difference in Boegel-EPII solutions when varying γ-glycidoxypropyltrimethoxysilane (GTMS) source between Dow Corning Z-6040 or Gelest SIG5840.0.
- Varying between the standard mixing procedures (Table 1) and “simplified mixing instructions” (section 3.5.2) did not yield any differences in bond performance.
- Boegel-EPII solution can either be constantly stirred with a magnetic mixer or occasionally shaken during the 30-minute induction time without an effect on wedge test performance.
- Once mixed, Boegel-EPII solution can be used up to 24 hours without a loss in bond performance. However, Boegel-EPII solution appears “milky” after 24 hours of continuous mixing. To be safe, Boegel-EPII solution should only be used within 12 hours of mixing to ensure the solution is fresh and does not appear “milky.”

5.2 Chemat AL 9201 Solution Evaluation Results

Three separate types of processes were evaluated when using Chemat Technologies AL 9201 solution; an "overlap" process, a mixture process, and a grit-blast/silane-type process. In general, the bulleted list below summarizes the results obtained in the evaluation of AL 9201.

- Processes where AL 9201 solution and Cytec BR 6747-1 primer were applied in two separate steps did not yield cohesive failure modes in the wedge test.
- The process using a mixture of 30% (by weight) AL 9201 and 70% BR 6747-1 primer exhibited the best failure modes in the wedge test versus, significantly outperforming the two-step "overlap" processes.
- Cleaning grit-blasted surfaces with Brulin 815 alkaline cleaner prior to application of the AL 9201-BR 6747-1 mixture improved wedge test results versus removing residual grit with clean, dry, pressurized nitrogen. However, this required soaking in Brulin 815 with light air agitation and is not possible for on-aircraft use.
- Specimens prepared using the mixture process and bonded with Cytec FM 73M exhibited cohesive failures when tested at 120°F and 95-100% RH as well as 140°F and 95-100% RH.
- Specimens prepared using the mixture process and bonded with 3M Company AF 163-2M exhibited cohesive failures when tested at 120°F and 95-100% RH but exhibited mixed failure modes when tested at 140°F and 95-100% RH.

5.3 Comparison of Optimized Sol-Gel Processes to Other Standard Surface Preparations

A comparison between some of the user-friendly sol-gel surface preparations and existing field-level surface preparations for aluminum was conducted since the main goal of this program was to develop processes for on-aircraft bonded repairs. All specimens were fabricated from bare Al 2024-T3 and bonded with 3M Company AF 163-2M 0.06 psf film adhesive. The adhesive was cured for 60 minutes at 250°F and 35-40 psi in all cases.

PAA was conducted according to ASTM D 3933 and primed with Cytec BR 127 bond primer to a nominal thickness of 0.2 mil (0.0002 inch). The primer was cured for 60 minutes at 250°F after drying for 30 minutes at ambient temperature (70°F).

The P2 paste acid etch is a variation of the P2 acid etch tank process[25]. Inert silicon filler is added to the P2 acid and applied to the aluminum. Panels prepared with the P2 paste acid etch were cleaned with acetone and deoxidized with 3M Company Scotch-Brite™ 7447 All Purpose pads. P2 paste was applied to the bonding surfaces with an acid brush for 25 minutes. After the 25-minute etch, the P2 paste was removed with a water rinse. The adherends were dried and primed with BR 127 bond primer to a nominal thickness of 0.2 mil (0.0002 inch). The primer was cured for 60 minutes at 250°F after drying for 30 minutes at ambient temperature.

Panels prepared with the grit-blast/silane (GBS) surface preparation[1] were cleaned with acetone and deoxidized with 3M Company Scotch-Brite™ 7447 All Purpose pads. Adherends were blasted with 50 μm Al_2O_3 grit. Residual grit was removed by blowing with 30-psi clean, dry, nitrogen. A 1.5% GTMS silane-water mixture was brush applied to the grit-blasted surface so the surfaces were wetted for 10 minutes. Residual silane was removed by blowing with 30-psi clean, dry, nitrogen until the panels were visibly dry. Panels were then placed in a preheated air-circulating oven for 60 minutes at 200°F. Once cooled, panels were primed with BR 127 bond primer to a nominal thickness of 0.2 mil (0.0002 inch). The primer was cured for 60 minutes at 250°F after drying for 30 minutes at ambient temperature.

Personnel at the Naval Aviation Depot in Jacksonville, FL, prepared the grit-blast/Pasa-Jell 105 specimens. The specimens were primed via brushing with BR 127 bond primer. All panels were sent to AFRL for bonding and testing.

The nylon pad/silane surface preparation was identical to the GBS surface preparation except the grit-blast step was removed from the process. Therefore, the silane was applied to the Scotch-Brite™-abraded surface. The reason for including this process was to compare the results to nylon pad/Boegel-EPII.

Panels prepared with the grit-blast/Boegel-EPII process and nylon pad/Boegel-EPII process were solvent degreased with acetone-soaked, lint-free cloths until clean. Adherends were deoxidized either with 50 μm Al_2O_3 grit-blast media or 3M Company Roloc™ medium nylon pads. Nylon pad abrasion was accomplished using a 20,000 RPM nitrogen-driven grinder. Once deoxidized, adherends were blown with clean, dry, compressed nitrogen to remove residual grit or debris. Boegel-EPII was applied with an acid brush within 30 minutes of the deoxidation process. The surfaces were kept wet for 3 minutes and the panels were orientated vertically to drain and dry under ambient conditions (70°F and 50% RH) for 30 minutes. Cytec BR 6747-1 bond primer was applied with a spray gun to a nominal thickness of 0.2 mil (0.0002 inch) and the adherends were dried at ambient temperature for 30 minutes. Primer was cocured with the adhesive for 60 minutes at 250°F and 35-40 psi.

Specimens prepared with the AL 9201/BR 6747-1 mixture process were solvent degreased with acetone-soaked lint free cloths until clean and then blasted with 50 μm aluminum oxide grit. Residual grit was removed by soaking adherends in Brulin 815 for 15 minutes using light air agitation at 140°F. Adherends were rinsed with DI water for 5 minutes and dried at 212°F for 30 minutes. A mixture composed of 70% BR 6747-1 and 30% AL 9201 by weight was stirred with a magnetic mixer for 10 minutes prior to use. Adherends were primed with the mixture to a thickness of 0.1-0.3 mil. The AL 9201/BR 6747-1 mixture was cured for 60 minutes at 250°F in an air-circulating oven prior to adhesive bonding.

Wedge test results, conducted at 120°F and 95-100% RH, are shown in Table 57. Specimens prepared with the Pasa-Jell 105 and nylon pad/silane processes exhibit failure modes less than 95% cohesive. All other surface preparations exhibit failure modes in excess of 95% cohesive. The two processes utilizing Boegel-EPII and the grit-blast process utilizing the Al 9201-BR 6747-1 mixture exhibit good failure modes after 28 days exposure at 120°F and 95-100% RH.

Table 57: A Comparison of Wedge Test Data at 120°F and 95-100% RH for Sol-Gel Surface Preparations versus Existing Surface Preparations on Al 2024-T3

Surface Preparation	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
PAA+BR 127	1.26	0.01	0.02	0.02	0.04	0.05	0.07	100% co
P2 Paste Acid Etch+BR 127	1.19	0.06	0.06	0.07	0.10	n/a	0.12	100% co
Grit-Blast/Silane+BR 127	1.21	0.07	0.07	0.10	0.13	n/a	0.15	100% co
Grit-Blast Pasa-Jell 105+BR 127	1.04	0.06	0.08	0.11	0.12	0.15	0.16	75% co
Nylon-Pad/Silane+BR 127	1.26	0.41	0.47	0.58	0.73	n/a	0.77	0% co
Grit-Blast/Boegel EPII+BR 6747-1	1.17	0.03	0.05	0.09	0.09	0.15	0.15	97% co
Nylon-Pad/Boegel EPII+BR 6747-1	1.07	0.02	0.08	0.12	0.16	0.16	0.20	95% co
Grit-Blast+AL 9201/BR 6747-1 Mixture	1.18	0.03	0.06	0.11	0.13	0.15	0.18	95% co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

Results for wedge tests conducted at 140°F and 95-100% RH are shown in Table 58. Specimens prepared with the PAA and P2 paste acid etch exhibit 100% cohesive failure modes. GBS specimens exhibit complete adhesive failure. Pasa-Jell 105 specimens exhibit mixed failure modes. When comparing these results to sol-gel specimen results, it is seen that the grit-blast/Boegel-EPII specimens exhibit approximately 95% cohesive failure modes. Specimens prepared with the AL 9201/BR 6747-1 mixture also exhibit high percentages of cohesive failure. The nylon pad/Boegel-EPII process yielded failure modes around 86% cohesive. Although these results are inferior to those obtained using PAA and P2 etch, they surpass GBS and Pasa-Jell 105 when tested at 140°F. It should be noted that longer crack lengths observed in the Boegel-EPII-prepared specimens were likely due to the presence small "nicks" of adhesive failure at the specimen edges. Since the edge of the specimen is where crack length is measured, the overall specimen crack length is measured to be worse than a completely cohesive specimen such as PAA even though the specimens with "nicks" have nearly 100% cohesive failure.

Table 58: A Comparison of Wedge Test Data at 140°F and 95-100% RH for Sol-Gel Surface Preparations vs. Existing Surface Preparations on Al 2024-T3

Surface Preparation	Initial (in)	Cummulative Crack Growth (in)						Failure Mode*
		1 hr	8 hr	24 hr	7 days	21 days	28 days	
PAA+BR 127	1.12	0.03	0.04	0.04	0.06	0.09	0.10	100% co
P2 Paste Acid Etch+BR 127	1.18	0.07	0.10	0.13	0.15	n/a	0.21	100% co
Grit-Blast/Silane+BR 127	1.08	0.06	0.13	0.15	0.22	n/a	0.32	0% co
Grit-Blast Pasa-Jell 105+BR 127	1.08	0.07	0.08	0.10	0.15	0.19	0.21	60% co
Grit-Blast/Boegel EPII+BR 6747-1	1.09	0.01	0.14	0.14	0.15	0.24	0.27	95% co
Nylon-Pad/Boegel EPII+BR 6747-1	1.09	0.05	0.11	0.15	0.22	0.29	0.33	86% co
Grit-Blast+AL 9201/BR 6747-1 Mixture	1.13	0.04	0.05	0.13	0.18	0.22	0.23	94% Co

* co: cohesive failure
Remaining non-cohesive failure occured betweeen the primer and aluminum

6 CONCLUSIONS

Two sol-gel chemistries were evaluated for bonded repair processes on aluminum alloys, Chemat AL 9201 and Boeing's Boegel-EPII. Adhesively bonded specimens were fabricated with both of these sol-gel solutions and evaluated for moisture durability using the wedge test.

Chemat Technologies, Inc.'s AL 9201 sol-gel solution appeared to provide good bonded joint performance, as measured by the wedge test, only when used as a mixture with Cytec BR 6747-1 primer. The process utilizing the AL 9201/BR 6747-1 mixture exhibited wedge test performance comparable to that of currently used surface preparations such as grit-blast/Pasa-Jell 105 and GBS. However, in order to obtain the best results with the mixture process, several inconvenient steps were required, including a Brulin 815 cleaning step after grit-blasting and a 30-minute elevated temperature drying step. The step is impractical for on-aircraft repair scenarios and therefore difficult to transition into a field-usable process. Unlike the processes utilizing Boegel-EPII, there was not a significant amount of application time saved utilizing the AL 9201/BR 6747-1 mixture process. The AL 9201/BR 6747-1 mixture may be useful in original equipment manufacturing since the processing steps are more suited for an automated line versus field-level maintenance and the mixture would have a long pot life, based on data for the individual constituents. Close cooperation between Chemat and Cytec would be required to field this approach.

The use of Boegel-EPII coatings on aluminum has produced strong and durable adhesive bonds when used in conjunction with several deoxidation techniques including grit-blast, nylon pad abrasion, sandpaper abrasion, and laser deoxidation. Grit-blasting appeared to provide a greater level of durability than the nylon pad and sandpaper abrasion techniques. Laser deoxidation appears to be another promising deoxidation method for laboratory tests although additional testing is required in order to develop a field-usable process.

Grit-blast and nongrit-blast processes utilizing Boegel-EPII yielded wedge test results comparable to those of several existing surface preparations currently used for aircraft bonding applications, including P2 paste acid etch, grit-blast/Pasa-Jell 105, and grit-blast/silane (GBS).

In fact, wedge test data from Boegel-EPII processes exceeded the performance exhibited by the grit-blast/Pasa-Jell 105 and GBS processes when tested at 140°F and 95-100% RH. Both the grit-blast/Pasa-Jell 105 and GBS processes are currently used for on-aircraft bonded repairs. Along with adequate bond performance, the surface preparations utilizing Boegel-EPII provide several other benefits. Boegel-EPII is more environmentally friendly and less of a safety hazard than the strong acids currently used in a number of alternate surface preparations. Since the Boegel-EPII solution has a pH greater than 4, there is less chance of hydrogen embrittlement of high-strength steel fasteners or corrosion concerns on aging aircraft structures. The Boegel-EPII surface preparations are also simpler to perform and take less time to apply than the currently used GBS process since the Boegel-EPII sol-gel processes do not require separate heating cycles for the silane, primer, and adhesive. Boegel-EPII is dried on the metal surface at ambient temperature without rinsing, and the primer and adhesive can be cured with a single elevated-temperature cure cycle. Since two separate cure cycles can be eliminated from the repair process, a minimum of 2.5 hours can be saved versus GBS while actually improving bond performance.

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ADDITIONAL REPORTS AND PAPERS

Several other papers and reports were generated under this contract. This section lists those publications for further reference.

- D.B. McCray and J.J. Mazza, "Optimization of Sol-Gel Surface Preparations for Repair Bonding of Aluminum Alloys," Science of Advanced Materials and Process Engineering Series, Volume 45. Society for the Advancement of Materials and Process Engineering. Pages 45-56, May 2000.

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